METALLOGRAPHERS' HANDBOOK OF ETCHING

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FOREWORD

At the Autumn Meeting of the Society of Swedish Metallographers in September, 1926, Mr. G. Malmberg, then Vice-President, put forward a proposal for the publication at the Society's expense of a *Handbook of Etching* for iron and steel. The proposition was adopted, and at the Spring Meeting in 1927 it was resolved that the necessary work should be commenced as soon as possible.

Mr. B. Palmgren, metallurgical engineer, was appointed to carry out the task and, at the Institute of Metallography in October, 1927, he began to search through the more recent literature (from the beginning of 1916 onwards). At the end of November, however, he was prevented from continuing, after having gone through *Stahl und Eisen*, 1916–27, the *Journal of the Iron and Steel Institute*, 1916–27, and the *Révue de Metallurgie*, 1916–22, together with smaller sections of other periodicals.

An arrangement was then made with Mr. T. Berglund, metallurgical engineer, to complete the collection of data and to correlate the material.

As the work proceeded it appeared advisable—in accordance with expressed requests—to enlarge the original programme to include also such non-ferrous metals as are met with in an industrial laboratory. The additional searching of the literature which was thereby rendered necessary has, of course, delayed the completion of the work.

In order to be able to utilize in the handbook the experience with etching reagents which has been acquired in many industrial organizations, a circular was sent out in January, 1928, to eighteen iron-works and laboratories. Of the thirteen replies received seven contained a number of valuable facts which have been duly incorporated in the book.

The Society of Metallographers wishes to express here its gratitude to those firms which, in the above way, have helped to increase the value of the handbook.

The finished manuscript has been read completely through by the Society's secretary, Mr. G. Phragmén, who also compiled a large part of the general account of etching reactions; in addition, there are certain sections by the present writer. Furthermore, copies of the manuscript were sent to, and criticized by, various members of the Council and some other interested parties. The Council hopes, therefore, that the work will not be found to contain any serious errors, and that it will serve its purpose for the benefit of industry.

Finally, the Council would appreciate any criticisms of the handbook and suggestions for its future improvement. These should be sent to Metallografiska Institutet, Stockholm.

For the Society of Swedish Metallographers,

CARL BENEDICKS,

President.

TRANSLATOR'S PREFACE

As regards the object of this Handbook there is little to add to what is said in the Foreword and Introduction. The book is addressed primarily to the many metallographers in industry who are often handicapped in their work by being unable to consult large technical libraries when requiring information on etching reagents and methods.

It will be found that the present volume is thoroughly practical in its outlook and that, as far as possible, no relevant detail regarding the etching reagents described or their manipulation has been omitted. From this point of view, therefore, it is hoped that the book will also be of use as a laboratory manual for advanced students of the science of metals.

In order to save space in the text the initials A.S.S.T. and A.S.T.M. have been used instead of the somewhat lengthy titles of the American Society for Steel Treating and the American Society for Testing Materials respectively.

I wish to express my thanks to Messrs. Chapman and Hall, Ltd., for their kind permission to include Tables VII and VIII.

It is also a very great pleasure to be able to thank Mr. Torkel Berglund and Mr. Helge Löfquist for their kindness in reading and criticizing the manuscript of the translation, and also Dr. Carl Benedicks for his continued personal interest and help throughout.

W. H. D.

HENDON.

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METALLOGRAPHERS' HANDBOOK OF ETCHING

CHAPTER I

INTRODUCTORY

> " Etching provides an easy way of distinguishing the different kinds of iron and steel, with regard to their hardness, density, and equality or inequality of intrinsic properties. . . ."
>
> SVEN RINMAN, 1774.

THE etching of iron and steel with acids in order to render visible the structure of the material has been employed for a very long time. As early as 1773 Sven Rinman[1] recommended such etching for distinguishing between real and imitation damascening; the words quoted above were written by him in 1774.[2] N. G. Sefström[3] stated in 1825 that "for demonstrating the uniform texture of bar-iron, this etching or corrosion with acids is a good method."

Widmanstätten in the year 1808 demonstrated the structure of ground and polished meteoric iron by etching with acids, and by heat-tinting.[4]

As practised, these early methods gave typical macro-etchings, and it was only when the microscope came into wider use for the examination of ground and polished sections of metals that the pioneers of metallography, Sorby, Martens, and Osmond, carried out tests on a number of micro-etching reagents for iron and steel.[5]

The first real period of development of metallurgical microscopy occurred during 1870–80, and at this time etching was carried out with very dilute aqueous solutions of acids, particularly nitric acid and hydrochloric acid, and with iodine dissolved in alcohol supplemented by the methods of "relief polishing" and "polish attack" (Osmond), and of "heat tinting" (Martens and Behrens). In the years immediately following, the technique of microscopical investigation and, naturally, of etching developed very rapidly. Among the investigators who improved etching methods were Wedding, Heyn, Le Chatelier, Stead, Ischewsky, Kourbatoff, and those already mentioned.

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[1] *Vetenskaps Akademiens Handlingar*, Vol. 34 (1773), p. 318.

[2] *Vetenskaps Akademiens Handlingar*, Vol. 35 (1774), p. 14

[3] *Jernkontorets Annaler* ,1825, p. 155.

[4] F. Osmond, *Cristallographie du Fer*, Paris, 1900, p. 25.

[5] The first communications by H. Sorby are found in *Proc. Sheffield Lit. Phil. Soc.*, 1864, and *Brit. Assoc. Report*, 1864, II, 189.

Thus by about the year 1905 most of the types of etching reagent used to-day had been tested, but subsequently their compositions have been individually modified and improved in order to meet increased demands on their selectivity and power of definition. Also, as the years have gone by, new kinds of steels and alloys have come into use, and in their turn created a need for new etching reagents.

The large numbers of etching reagents and etching methods which have been proposed from time to time have led to the result that the metallographer, when concerned with the etching of an uncommon or new material, will often hesitate in his choice of a suitable reagent.

Previous Compilations. It is true, of course, that there already exists a number of very useful collections of the etching reagents in most general use, together with descriptions of their chief effects, but all these are either incomplete and condensed, or deal only with certain limited fields.

Among the most important of these special collections may be mentioned the following:—

For Iron and Steel.

R. S. Kerns, The Foundry Data Sheets, Nos. 335–338. Supplement to *The Foundry*, 1st and 15th June, 1920.

E. C. Groesbeck, Bureau of Standards, *Scientific Paper No.* 518, 1920. (Alloy Steels.)

Werkstoff-Handbuch, Stahl und Eisen, Blatt **VII**–7, **VII**–15, 1927.

American Society for Testing Materials, *Standards,* 1927, I, p. 756.

American Society for Steel Treating, *Handbook,* 1929, p. 325.

FOR NON-FERROUS METALS.

J. Czochralski, Stahl und Eisen, 1915, pp. 1072 and 1129. (Various Metals.)

H. S. Rawdon and M. G. Lorentz. Bureau of Standards, *Scientific Paper No.* 399, 1920. (Cu.)

H. S. Rawdon and M. G. Lorentz. Bureau of Standards, *Scientific Paper No.* 435, 1922. (Cu-alloys, Ni and Ni-alloys.)

Werkstoff-Handbuch, Nichteisenmetalle, Blatt C13–14, 1927. (Various.)

American Society for Testing Materials. *Proceedings,* 1927, I., p. 769. (Various.)

Bureau of Standards, *Circular No.* 346, 1927, p. 21. (Aluminium.)

American Society for Steel Treating, *Handbook,* 1929, p. 325. (Various.)

The aim of the present work has been to collect into a handy reference book all information published to date which seemed to be of value. In so doing an attempt has been made to develop the subject as systematically and concisely as possible, while preserving the essential details given in the various original articles. Thus, all information regarding the manufacture of etching solutions, their use, etching time, etc., is given in unaltered form, even if, as a result, the presentation as a whole becomes somewhat disconnected. In so far as it has been possible and convenient the chronological sequence of the various reports has been followed. As a result of this procedure certain ideas can sometimes be obtained for experiments with new etching reagents or modifications of old ones.

Occasionally an etching reagent has been specially tested, and the results obtained reported.

In the majority of cases, however, the published information on the effect of an etching reagent has, of necessity, been given without criticism. Nevertheless in more than one instance, particularly in regard to non-ferrous metals, the description of the effect of etching has been based on far too slight a knowledge of the constitution of the alloys concerned. An example of how a mistake can arise through the uncritical use of etching reactions is to be found in a well-known work on iron-chromium alloys[1] (*cf.* Westgren, Phragmén, and Negresco).[2] Sometimes errors are caused by insufficient attention being paid to the differences in etching reactions caused by variations in the compositions of the solid solutions present.

When using this handbook, therefore, a certain critical attitude is necessary, since had criticisms been incorporated in the book itself it would have expanded into a textbook of metallography.

The inclusion of systematically chosen photographs illustrating the action of the etching reagents would have been an advantage, but they occur very rarely in the literature. A few explanatory photographs have, however, been inserted by the writer.

It is clear that a more scientific study of the action of etching reagents would be of great value. Actually, there are scarcely any potential measurements from the standpoint of etching to be found in the literature. Nevertheless an experimental investigation for the elucidation of such problems is outside the scope of the present work, and must be deferred to the future.

General Arrangement. In the following pages the subject-matter is grouped into one general, and three special, chapters which are followed by a bibliography.

The general chapter deals with the most essential points in the preparation of the specimens, and with the general properties of etching reagents, and also gives an outline of etching technique. The subject of the preparation of specimens has been dealt with very briefly, since it must be considered to be somewhat beside the point in a collection of information on metallographic etching. The special chapters deal respectively with the macro-etching of iron and steel, the micro-etching of iron and steel, and the etching of non-ferrous metals.

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[1] *Journal of the Iron and Steel Institute*, 1919, *100*, 445.
[2] *Ibid.*, 1928, *117*, 383.

The bibliography is divided into two parts, the first of which contains all the papers, etc., referred to in the text, while in the second some additional references are grouped under various headings. In all cases the references are given under authors' names, and the abbreviations for the Journals are those adopted by the Institute of Metals, London.

CHAPTER II

GENERAL CONSIDERATIONS

Sampling. When investigating the properties of a material, the first thing to do is to obtain one or more suitable samples. For metals, just as for other substances, it is essential that specimens intended for examination should be taken in such a way, and be of such a size, that they are truly representative of the material from which they are removed. This holds both as regards the chemical composition of the material and its physical condition. The specimens should, therefore, be cut in such a manner that the material does not change its character as a result of heating or cold-working.

The size chosen for the sample depends on the nature of the investigation, and on the mechanical resources available for its subsequent preparation. As regards its shape there should be, preferably, a definite relationship between the area of the grinding surface and the height: too great height is troublesome, and usually makes it difficult to obtain a flat polished section. For microscopical purposes grinding and polishing can be done most easily, and in the shortest time, on a section of one or two square centimetres area. The difficulties increase proportionally as it becomes necessary to deviate from this order of dimensions, and only for the most imperative reasons should a surface be prepared which exceeds about 6 sq. cms. in area. For microscopic investigations of such a kind that a finely ground surface is required, a section of area 1 sq. dm. may be considered as the upper limit for grinding by hand.

Mounting the Specimen for Grinding and Polishing. It is necessary to mount the specimen for grinding and polishing if it is too small to be conveniently held in the hand, and also if an examination of the edge of the section is important. For this purpose clamping, mounting in fusible material, or electroplating is employed.

Clamping is used for the examination of the cross-sections of thin sheets. The section is held between two metal straps held tightly together with at least two screws. Soldering is sometimes useful for holding them in position. Specially designed clamps are an advantage when dealing with many specimens of similar shape, which must be flat at the edges.

Mounting by casting a fusible material round the specimen (using a short piece of tubing as a mould) can be done with alloys such as Wood's metal or Babbitt metal, or with non-metallic materials such as sulphur, shellac, and dental-cement. Unalloyed lead is not, as a rule, suitable as a mounting medium because of its property of "smearing" during grinding. Wood's metal and Babbitt metal also smear, though to a less degree than lead. Shellac should only be used for hard metals because any particles detached during grinding will easily scratch a soft surface.

Electroplating is resorted to more rarely because the deposition takes up valuable time. The method is, however, invaluable in certain cases. For iron, the process consists of a preliminary copper-plating in a cyanide bath until the surface is thoroughly coated with a layer of the latter metal; the deposition is then continued in an acid bath in which it can be carried out more rapidly.

When etching a mounted specimen attention must be paid to the extent to which the mounting medium can have a disturbing effect on the process. It is safest, before etching, to remove any metallic mountings which may have been used. From this point of view mounting in sulphur is an advantage because it is electrically a non-conductor, and also insoluble in the majority of etching reagents.

Mechanical Preparation of the Surface. When preparing specimens by mechanical methods it should always be borne in mind that a cold-worked surface, in which the metal has "flowed," etches in quite a different manner from one which is free from mechanical deformation. The idea in preparing the specimen should be not only to obtain a smoother and more scratch-free surface at the end of each successive operation, but also to diminish similarly the thickness of the cold-worked layer on the surface. Only when this layer is so thin that it is entirely dissolved on etching does the true structure of the material appear.

Working with Cutting Tools. The surface of the specimen is worked first with a cutting tool, provided that no difficulties arise owing to hardness. Large surfaces for macroscopic examination are prepared by planing, turning, or milling. The surface so obtained is often sufficiently fine for the subsequent examination, especially if a sharp tool and a fine feed is used for the last cut. Smaller specimens which will subsequently be ground by hand, are preferably worked with a file, even if they have been previously planed by machinery. In this case the file is firmly fixed, and the specimen moved about on its surface. Second-cut files are particularly well adapted for this purpose, since the cut is such that the specimen does not tend to slip off during filing. The file should be first rubbed over with a piece of chalk to prevent the filings from sticking in the grooves.

For the softer metals, lead, tin, aluminium, copper, etc., preparation in powerful microtomes has recently come into use (Lucas, and others). By using these machines with properly set special knives, not only is the extremely difficult and tedious grinding of such materials rendered superfluous, but a better result is also obtained.

Grinding. Large surfaces can, with advantage, be ground mechanically in plane-grinding machines. If it is performed with adequate care, this method also provides a labour-saving preliminary preparation for small specimens of hard material. For abnormally large microsections it is the only one which can be economically used if a perfectly satisfactory specimen is required. In all machine grinding with rapidly rotating emery wheels heating must be avoided as far as possible. (Use light pressure, a suitable wheel, and liquid cooling.)

As regards the treatment of the specimens with emery-paper, etc., it must be emphasized that the condition for obtaining really good surfaces is to keep cold-working down to a minimum. When using a particular grade of the grinding material, therefore, it is essential not only to grind until the scratches due to the preceding coarser grade have disappeared, but also until the cold-worked layer produced thereby has been removed. This layer can, in the case of soft iron at least, be considered to be approximately equal in depth to the diameter of a grain of the grinding material which is being used. With a particular grade, therefore, it seems rational, having removed the scratches due to the preceding operation, to continue the grinding for as long a time again as was required to reach the latter stage.

Polishing. The final stage in the mechanical treatment of the surface of the specimen, that is, polishing, should, in the case of iron and steel and alloys of similar mechanical properties, not take more than five minutes at the most—assuming that it is done in the usual manner with purified alumina on a rotating cloth-covered disc. If the polishing takes longer, then the previous grinding has been done incorrectly, and a good surface cannot be obtained.

As far as the soft metals and alloys are concerned polishing often presents great difficulties, chiefly on account of their great tendency to "flow." These materials are usually polished with precipitated magnesia and distilled water on thin cloth. To be suitable for use the magnesia must have a fine grain-size, and not contain any agglomerated particles. An excellent test is as follows: a little magnesia is placed on the tongue from the point of a knife, and if, on gently rubbing the tongue against the roof of the mouth, the powder is felt to disappear rapidly and completely, the latter is suitable for use as a polishing medium.[1]

When polishing with magnesia the dry powder is sprinkled on the previously moistened cloth. A few more drops of water are added, and the magnesia then spread out over the cloth with the fingers. Any hard agglomerated particles felt during the process are removed. The polishing is done either entirely by hand or on a *slowly* rotating disc. The final polish is carried out on a well-washed cloth almost free from magnesia. Distilled, or well-boiled, water must be used because MgO easily combines with the carbonic acid in ordinary tap-water. The resulting magnesium carbonate forms large crystalline particles which produce deep scratches in the specimen. For the same reason the polishing-cloth must be carefully washed out immediately after use. It does no harm to wash it occasionally with a dilute solution of hydrochloric acid, containing 1–3 per cent HCl.

Etching the Specimen During Grinding. When it is a matter of considerable difficulty to obtain a surface reasonably free from cold-working, fairly deep etching with subsequent fine grinding or polishing is often of great help. The method is recommended by several authors (Archer, Bolton, and others).

Cleaning the Specimen After Polishing. After polishing, the specimen must be carefully cleaned from adhering polishing medium, and this is generally carried out in running water. The washing should be done as soon as possible after polishing, otherwise the surface has time to dry partially with the result that the polishing powder can only be removed with difficulty. After washing in water the specimen is, as a rule, rinsed with alcohol (dropped on the surface) and then, if the nature of the reagent to be used so permits, etched immediately without allowing the surface to dry. If a large number of specimens are being prepared for examination together, then, to save time, those already polished and washed can be conveniently stored for an hour or two under alcohol (preferably absolute) in a covered dish. In the majority of cases this process gives better results than if the specimens are dried before etching because, in the latter case, the surface has time to become slightly oxidized or to occlude gases, with the result that the etching is impaired. In this connection, however, it may be mentioned that in a few cases, to which attention will be drawn later, etching should definitely not be carried out immediately after polishing.

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[1] Recommended by F. F. Lucas, New York

On those occasions when the specimen has to be dried, this is done most simply if the specimen is washed quickly in running water, rinsed with a few drops of alcohol, and dried in warm air supplied, for example, from a hairdresser's fan. More efficient drying is achieved by the use of hot water and compressed air, e.g. from a cylinder. Of course, the specimen can also be dried with soft cloth, preferably linen, or chamois leather. Great attention should, however, be paid to the cleanliness of these materials, as otherwise they may easily do harm. The chamois leather should be washed with benzene or something similar before use, because the commercial article contains considerable amounts of grease.

The Purpose of Etching. In general a well ground and polished metal surface shows no trace of its structure because all incident light is reflected (or absorbed, as the case may be) in the same way over the whole of the polished area. Exceptions are provided, however, by those alloys which contain differently coloured phases, such as, for example, the alloys of copper and bismuth, of copper and copper oxide, or of aluminium and iron.

Etching is used to render visible the chemically or physically distinct parts, and its object is to produce, in one way or another, a more or less increased optical contrast between the various structural components. This is made possible by selective attack, or selective colouring, of the surface of the specimen. Both these etching effects often occur together.

A certain kind of "relief-effect" is obtained by purely physico-mechanical means—often quite unintentionally—when grinding and polishing, owing to the different resistances to abrasion of the various constituents. This mechanical formation of relief was formerly used to a very large extent as a process of "etching." Most of the etching methods now used, however, are based simply and solely upon chemical action on the polished metal surface, whether either a relief or a colour effect is desired.

The Chemistry of Etching. The etching reagents which are used in metallographic work are usually dissolved in water or ethyl alcohol or in mixtures of these liquids. In some cases, which are described subsequently, methyl alcohol, amyl alcohol, glycerine and acetone have also been proposed as solvents.

The free energy of the etching reactions, which must be regarded as ultimately determining the etching effect, is a function of the solvent, as the solution is not saturated with respect to all components. This function is, however, rather hard to define. An important factor is the velocity of the reaction, and this depends on the degree of dissociation of the reagent, and hence on the electrical conductivity of the solution, and also on the viscosity which, in turn, determines the rate of diffusion. It is, therefore, obvious that nitric acid, for example, will have quite different etching actions according as it is dissolved in water, ethyl alcohol, or amyl alcohol. The water-content of the alcoholic solutions is, therefore, of great importance.[1]

From a practical standpoint it should be noted that an aqueous solution wets a metallic surface much more unevenly than one in alcohol. This must primarily be due to the latter's property of dissolving grease. It is, therefore, easier to obtain even etching with alcoholic solutions.[2]

Acids are often used in etching reagents. Of these only HCl, HF, and H_2SO_4 really behave as acids, that is to say, according to a reaction represented by—

$$\mathrm{Fe} + 2\mathrm{H}^{\cdot} \rightarrow \mathrm{Fe}^{\cdot\cdot} + \mathrm{H}_2$$

which is due entirely to the presence of hydrogen-ions. The other acids employed have practically the properties of oxidizing agents. HNO_3 dissolves Cu, for example, in the following way—

$$3\ \mathrm{Cu} + 8\ \mathrm{H}^{\cdot} + 2\ \mathrm{NO}_3' \rightarrow 3\ \mathrm{Cu}^{\cdot\cdot} + 4\mathrm{H}_2\mathrm{O} + 2\ \mathrm{NO}$$

Chromic acid and picric acid behave similarly, as also does strong sulphuric acid at high temperatures.

Dilute solutions of HCl or H_2SO_4 do not, as is well known, attack Cu, with the evolution of hydrogen, but if some oxidizing agent, such as H_2O_2 or $(NH_4)_2\ S_2O_8$, is added, the Cu is dissolved thus—

$$\mathrm{Cu} + 2\ \mathrm{H}^{\cdot} + \mathrm{H}_2\mathrm{O}_2 \rightarrow \mathrm{Cu}^{\cdot\cdot} + 2\ \mathrm{H}_2\mathrm{O}$$

Even the oxygen of the air can be employed as the oxidizing agent, but then the reaction proceeds slowly.

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[1] This is well illustrated in an investigation by Benedicks and Sederholm (2). A slightly dissociated solution of 0·1 per cent HNO_3 in absolute alcohol causes the formation of an oxidized film which is partly protective against attack. On the other hand, with the more dissociated aqueous solution of the same strength nothing of the kind appears.

[2] Solutions in alcohol also have an advantage in that, because of their lower dissociation, they can be made considerably more concentrated than aqueous solutions without becoming too powerful in their action. Hence the same etching effect is obtained with a given portion of an alcoholic solution in spite of its being used many times, whereas a similar portion of the corresponding aqueous solution is very quickly spent. (See previous note.)

A solution of $(NH_4)_2 S_2O_8$ has a solvent action even without the addition of acid

$$Cu + S_2O_8'' \rightarrow Cu^{\cdot\cdot} + 2\,SO_4''$$

In some cases alkaline solutions are used for etching. Certain metals such as Al, Sn, or Zn, whose lowest oxides form amphoteric electrolytes, can be dissolved in these solutions with the evolution of hydrogen, while the metal itself forms anions.

$$2\,Al + 6\,OH' \rightarrow 2\,AlO_3''' + 3\,H_2$$

The solution of Cu in ammonia is of quite another kind. Cu does not dissolve unless some oxidizing agent is present. Oxygen from the air is sufficient but, usually, H_2O_2 is added, and the reaction then proceeds thus—

$$Cu + 4\,NH_3 + 2\,H^{\cdot} + H_2O_2 \rightarrow Cu\,(NH_3)_4^{\cdot\cdot} + 2\,H_2O$$

That is, the copper forms cations and not anions.

The oxidizing agents mentioned above all have a relatively high reaction potential and, furthermore, have the property of evolving oxygen directly. Hence, when used in the etching of chromium alloys, for example, they easily produce passivity. Now the oxidation of a metal really consists of each atom losing one or more electrons, and may be represented thus

$$Fe \rightarrow Fe^{\cdot\cdot} + 2$$ [illegible]

If this is carried out by making the metal the anode in a suitable electrolyte, the reaction potential can be perfectly controlled by varying the current density.

$Fe\,Cl_3$ is often used as a weak oxidizing agent. When etching Cu, for instance, the reaction is of the type—

$$Cu + 2\,Fe^{\cdot\cdot\cdot} \rightarrow Cu^{\cdot\cdot} + 2\,Fe^{\cdot\cdot}$$

It proceeds without alteration of the acidity of the liquid, and hence hydrogen ions do not enter into the equation. At the same time, however, the concentration of Cl-ions will have some influence, owing to the formation of complex ions of $CuCl_3'$. [The solution should, of course, be slightly acid to prevent hydrolysis of the $Fe\ Cl_3$.]

$CuCl_2$ also behaves in much the same way as $Fe\ Cl_3$. In the etching of iron the reaction is represented by—

$$Fe + Cu^{\cdot\cdot} \rightarrow Fe^{\cdot\cdot} + Cu$$

That is, Cu is precipitated on the iron. If, however, HCl is added in sufficient quantity the deposition of Cu is prevented, and the reaction then proceeds thus—

$$Fe + 2\ Cu^{\cdot\cdot} \rightarrow Fe^{\cdot\cdot} + 2\ Cu^{\cdot}.$$

Since hydrogen ions do not enter into the reaction the acidity of the solution should not influence the equilibrium, but here again the Cl-ion concentration will have an effect. Cu-ions can form complex groups according to the equation—

$$Cu^{\cdot} + 3\ Cl' \rightarrow Cu\ Cl_3''$$

and in this case the equilibrium is displaced much further to the right than in the reaction—

$$Cu^{\cdot\cdot} + 3\ Cl' \rightarrow Cu\ Cl_3'.$$

Therefore, for solutions with a high concentration of chlorine ions the equation for the etching reaction should be written—

$$Fe + 2\ Cu^{\cdot\cdot} + 6\ Cl' \rightarrow Fe^{\cdot\cdot} + 2\ Cu\ Cl_3''.$$

In this discussion only aqueous solutions have been considered, but in practical work with copper-containing etching reagents, mixtures of alcohol and water are usually employed as solvents. These will have a marked effect on the dissociation constants, but in point of principle the reactions remain the same.

Besides the oxidizing agents mentioned above, free halogens can also be employed occasionally as etching reagents. Iodine may be used in alcohol, or in an aqueous solution of KI. Bromine and chlorine can be used in solution in water or alcohol. The solutions need not be acidified (unless with the ultimate object of preventing the precipitation of basic salts) The equations for the reactions are of the type

$$2\ Fe + 3\ I_3' = 2\ Fe''' + 9\ I'$$

As regards electrolytic etching it may be pointed out that, as long as the reaction at the anode implies only the charging and detaching of the metal atoms as ions, the nature of the electrolyte will have no sensible influence provided that its conductivity is sufficiently high. The nature of the anions decides the maximum voltage at the anode, and will also be of importance if any secondary reactions occur there. On the other hand, the nature of the cations should be of no consequence; hence it is difficult to explain, say, the effect which in some cases is ascribed to the addition of $Fe\,Cl_3$.

The hydrogen-ion concentration of the solution, however, exerts some influence because the reaction at the cathode frequently tends to make the solution less acid.

Grain-contrast and Grain-boundary Etching. When the specimen consists of more than one phase, the various phases are generally attacked with different intensities. The more strongly attacked areas usually become rougher and, therefore, appear darker, and in some cases it may happen that one phase is coloured by the products resulting from the reaction.

On the other hand, when the alloy consists of only one phase the various grains will, after etching, usually appear to be of different degrees of brightness. In most cases this is due to the formation of pits bounded by surfaces which approximate to planes with simple crystallographic indices,[1] i.e. the so-called etch-figures long known to mineralogists. Since the "facets" so formed are arranged in parallel groups in each crystal, the light will be reflected in the same way from each surface element belonging to the same crystal. In a neighbouring grain, where the crystal axes have another direction, the light will be reflected in a different manner. The roughening of the surface may be so slight that it is hardly noticeable, even at the highest magnifications. The most striking feature in this kind of grain-contrast etching is that the relative brightness of the grains is dependent in the highest degree on the direction of the incident light.

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[1] J. A. Ewing and W. Rosenhain. *Phil. Trans. Roy. Soc.* (*A*), 1899, *193*, 352.

If the etching is carried out in such a way that the etch figures become sufficiently large to be studied individually, the process may be described as "crystal figure etching" (Czochralski). An example is given in Fig. 1.

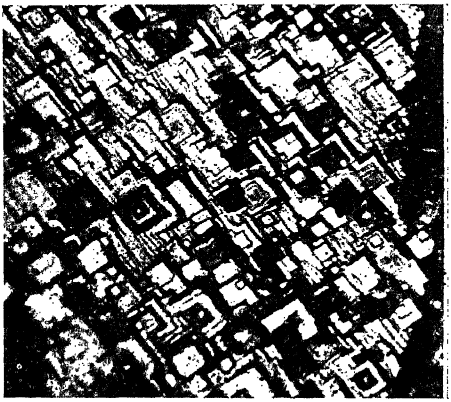

FIG. 1. "CRYSTAL FIGURE" ETCHING OF COPPER
Etched with 10% ammonium persulphate. × 200. (Czochralski.)

The orientation of the crystal axes within the grain determines not only the shape of the etch figures but also the speed with which solution occurs. A typical example occurs in the etching of pure iron with picric acid. The specimen remains tolerably bright, but a network of lines becomes visible. Subsequent etching with ammonium persulphate shows that these lines which become visible the earliest are the grain boundaries. A critical examination at high magnification with oblique illumination shows that the appearance of grain-boundaries is due to differences of level between the various grains.

This variation in speed of reaction can also produce a difference of colour from grain to grain if, as a result of the etching action, coloured and more or less insoluble products are formed. This is probably the case when etching soft iron with nitric acid in amyl alcohol. In this way a grain-contrast etching is obtained which is of a different type from the one mentioned above.

The grain-boundaries can also be made visible in another way which occurs when, as in most cases, the attack of the etching reagent is most rapid at the edges of the grains themselves, thus forming a deep channel between them. An appropriate example can be obtained by etching soft iron with nitric acid in ethyl alcohol.

The characteristic of grain-contrast and grain-boundary etching is that the result is governed by the directions of the crystal axes within the various grains.

Primary Etching. On account of the phenomenon of segregation which accompanies solidification, some difference in chemical composition nearly always occurs between the first and last portions of an alloy to freeze. With a suitable etching reagent this chemical heterogeneity can be made visible, even in those cases where only one phase occurs, such as in copper-rich brass or bronze, or in iron with a moderate percentage of phosphorus.

The chemical heterogeneity caused by the process of solidification should not, of course, be confused with the heterogeneity caused by subsequent polymorphic changes.

By subsequent treatment it is certainly possible to level up the differences somewhat, but they can seldom be entirely eliminated. Hence, by investigating these heterogeneities it is possible to get an idea of the general distribution of the crystals formed on solidification, even if the crystal grains actually existing in the specimen have entirely different orientations as a result of polymorphic transformations or recrystallization.

For this reason Oberhoffer (3)[1] has denoted by the term "primary etching" an etching which brings out the chemical heterogeneity. (See Fig. 2).

This term is now in common use, but the analogous expression "secondary etching," also proposed by Oberhoffer to include grain-contrast and grain-boundary etching, is not so satisfactory as it assumes that some transformation or recrystallization has taken place.

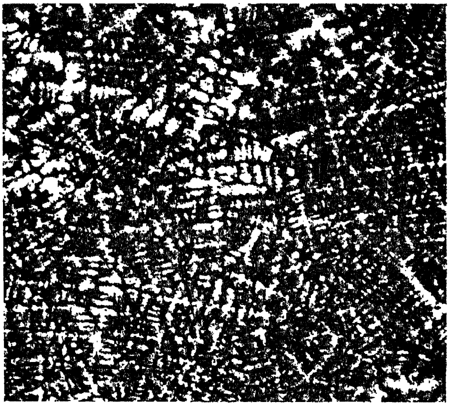

FIG. 2. PRIMARY ETCHING

Cast crucible steel, containing 0·35% C, 3·25% Ni, 0·75% Cr, 0·09% P. Oberhoffer's etching reagent. × 10.

As a rule an etching reagent does not produce only a grain-contrast (or boundary) etching, or only a primary etching, but tends to give both effects together. However, by choosing a suitable reagent one or the other type of etching can be caused to preponderate.

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[1] This and similar numbers in the text refer to the appropriate entries in the bibliography under the author's name quoted.

Some Factors Influencing the Result of Etching. Besides the chemical natures of the specimen and of the reagent, the other factors which exert an influence on the result of etching are the mechanical condition of the surface of the specimen, the time of etching, the temperatures of the reagent and the specimen, and the agitation of the reagent during etching.

It has already been pointed out that the cold-working of a metal affects its properties to a very great extent. With increasing degrees of cold-working the direct reflection of light after normal etching becomes less and less, and is replaced ultimately by a diffuse reflection which is independent of the direction of the incident light.

A metal surface in the condition in which etching develops only a general matt appearance does not show any grain structure; only constituents differing in their chemical compositions can be seen, e.g. cementite and ferrite in severely cold-rolled steel.

In the case of soft materials, in which a slightly cold-worked surface can hardly be avoided when preparing the specimen, it is sometimes advisable to heat the latter slightly so that the strains present in the surface layer may have an opportunity of being released. Of course, this heating must be done with due care, so that the structure of the specimen itself is not altered. Thus, when preparing specimens of soft iron,[1] the subsequent appearance on etching of "latent" grinding scratches can be partly prevented if the specimen, after treatment in the ordinary way, is warmed up in an oil-bath to 200–300° C. for an hour or two, and subsequently subjected to a light but thorough repolishing. To a certain extent the same effect may be obtained by storing the specimen for some considerable time after finishing on the polishing wheel.

As regards the effects of time and temperature on etching, Loskiewicz has made a number of investigations on the influence of these factors in the etching of carbon steel in nitric and picric acids. The time was measured up to the point when the lamellar structure of the pearlite, and the grain boundaries of the ferrite could be observed clearly and simultaneously, while the surfaces of the ferrite grains remained practically unattacked.

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[1] Author's investigation at the Bureau of Standards.

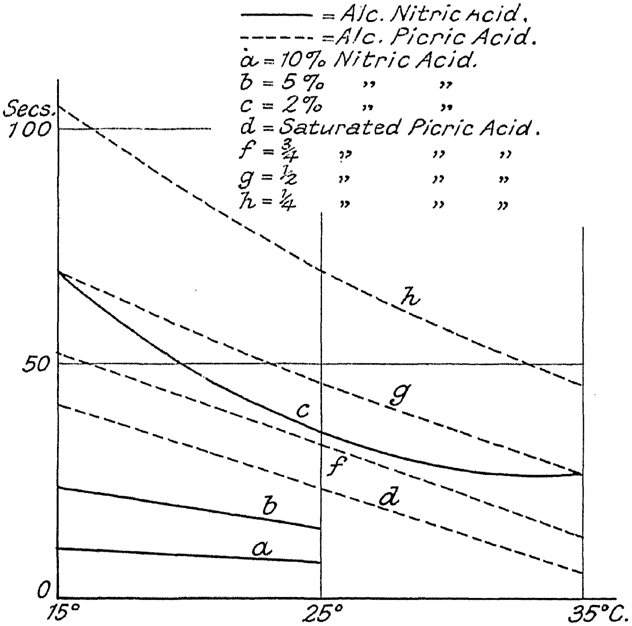

Fig. 3. The Effect of Temperature on the Etching Time of Pearlite in 0·30 per cent Carbon Steel (Loskiewicz)

The time interval required to obtain the best etch at a definite temperature was found to vary in magnitude with the concentration of the reagent. In the case of a material containing 0·3 per cent carbon, the interval had its greatest values for 5 per cent nitric acid, and ¼-saturated picric acid, both in ethyl alcohol. The time in which a good etch is obtained is 15–30 seconds for nitric acid, and 80–125 seconds for picric acid. Fig. 3 shows the relation of the etching time to the concentration and temperature of the reagent. (Only the lines through the average values are shown.) The time of etching is approximately a linear function of the temperature of the reagent. At 15° C. etching takes almost twice as long as at 35° C.

As regards Loskiewicz' results it may be mentioned that the etching effect of nitric acid depends on the temperature to a much greater extent than appears from the above. At low temperatures nitric acid behaves purely as a solvent, and gives grain-boundary etching, but at elevated temperatures it acts as an oxidizer and produces grain-contrast etching. The transition temperature between these two modes of action lies at ordinary room temperature (*cf.* page 84).

Agitation of the reagent during etching considerably affects the speed of the process, and sometimes even the result. Thus, when etching with alcoholic picric acid, it will be observed that, in the absence of agitation, the surface of the specimen is soon darkened by a layer of more or less insoluble products which retard the attack. By moving the specimen about during etching these reaction products are removed, so that the surface is kept cleaner and the process proceeds more rapidly.

Etching Dishes and Specimen Holders. It is preferable to use glass or porcelain dishes as containers for reagents during etching. Crystallizing dishes with overhanging covers are probably the most useful. When macro-etching large specimens a paraffined wooden trough is often employed.

In the case of hydrofluoric acid and its solutions the bottoms of the paraffin flasks in which the acid is usually sold can be used with advantage. Paraffined glass dishes may also be employed and, of course, the graduated vessel for measuring out the strong acid should be suitably waxed. Very dilute solutions of hydrofluoric acid can be used in ordinary glass vessels without any trouble, but, in such cases, the solutions should be freshly prepared for use.

Metal dishes of platinum, etc., should be used with care because of the possibility of electrolytic action. Boxes made of folded lead sheets make excellent containers when etching large specimens with hydrofluoric or sulphuric acid.

During etching the specimen is held either with rubber-gloved fingers or with an instrument made preferably of some electrically insulating material such as wood, ebonite, rubber, or porcelain. Cheap and simple tweezers can be easily made from wood. (Clothes pegs are sometimes very useful in this connection.) Metal pincers and tongs can be used in the majority of cases without inconvenience, but care must be taken that no local elements are set up to disturb the etching. It is for this reason that a specimen has often to be removed from the metal in which it has been mounted for grinding and polishing.

Crucible tongs made of nickel-chrome are excellent for use when etching.

Etching by Immersion. The simplest and commonest method of etching is by immersion. A quantity of the etching solution is poured into a dish suited to the size of the specimen, and the latter, by means of a holder, put in with the polished surface uppermost and so that it is entirely covered by the solution. In most cases it is desirable to keep the liquid in motion during etching, so as to prevent the occurrence of local variations in concentration which may cause changes in the normal working of the reagent. This can be done by allowing the specimen to rest on the bottom of the dish while imparting a rocking or swirling motion to the whole, or by moving the specimen about with the holder in the stationary dish. Occasionally it may be advisable to let the specimen rest on the bottom of the dish and, during the etching period, to brush over the surface with a soft hair-brush or with cotton-wool which has been wrapped firmly round the end of a wooden stick. The so called "orange sticks" (obtainable from chemists) are very convenient for this purpose. Brushing is particularly useful when dealing with an etching reagent which deposits more or less insoluble products of the reaction on the metal concerned. As mentioned previously, an example of this occurs in the etching of iron with an alcoholic solution of picric acid.

Drop by Drop Etching. In this method the polished surface of the specimen is held in an horizontal position and as much of the solution as it can hold by surface tension is dropped on to it most conveniently from a dropping bottle. This method has the advantage of requiring only small quantities of etching liquid, but it is only suitable for those cases in which the time of etching is short. The condition for the method to be successful is that the specimen must be completely and thoroughly dried before the solution is dropped on to it.

Wash Etching. This method is often used for microsections when a suitable etching dish is not available, and the drop-by-drop method does not give a satisfactory result.

The surface is held upwards, slightly inclined, and the etching solution is supplied to the highest edge from a dropping bottle, and allowed to wash down over the surface.

The process is sometimes used on a large scale when macro-etching ingot sections, for example, for which no satisfactory container can be found, or when it is desired to confine the attack to the ground surface only.

Etching by Dipping. A combination of the three foregoing methods results in one which may be called etching by dipping (recommended by Gat).

The specimen is held in tongs with the polished surface downwards, and dipped just beneath the surface of the etching liquid contained in a dish. Then, after remaining there only a relatively short time, it is lifted out and the tongs reversed so that the surface becomes uppermost. The latter is watched, and the process is repeated if necessary until a satisfactory etch is obtained. In this method, as in the previous one, the oxygen of the air has a good opportunity of taking part, and this is sometimes important as regards the etching effect.

Etching by Swabbing. In this process the surface of the specimen is rubbed more or less vigorously with a wad of cotton-wool which has been soaked in the etching solution and lightly squeezed out. The correct pressure to apply depends on the purpose of the rubbing and on the possibility of the surface being damaged by scratches. This procedure is used fairly often when the etching reagent tends to develop on the surface of the specimen some kind of adhesive film which has to be removed in order to obtain a uniformly progressing attack. If the film which is formed adheres tightly greater pressure is, of course, required than if it remains loose. When necessary the wad of wool is again saturated with the solution. In this method of etching the oxygen of the air sometimes plays a definite part.

Polish Attack. Osmond's method of polish attack is really a more intense form of swabbing, except that the air has less opportunity to act on the surface. A convenient amount of the etching medium is spread on cloth, rubber, or saturated parchment, and the specimen given a second polishing on it, preferably by hand. The result is a combined etching and relief-polishing effect.

The more elastic is the support, the more pronounced is the relief effect.

Relief Polishing. Relief polishing, which was also introduced by Osmond, is carried out like the above process on an elastic support but instead of an etching reagent only water is used, together with a suitable polishing medium, such as rouge.

Electrolytic Etching. Electrolytic etching to develop the structure by anodic solution was probably first used by Le Chatelier in 1896.

The method is particularly useful for those metals and alloys, such as high-alloy steels, nickel chrome alloys, stellite, etc., which have a special resistance to attack by chemical reagents. Metals and alloys which easily become passive on anodic treatment must generally be activated first by means of a relatively powerful cathodic treatment.

For electrolytic etching it is most convenient to use a platinum dish as the electrolyzing vessel. The specimen, with the polished surface upwards, is placed inside and in metallic contact with the dish which is connected to the positive pole of the current supply. The cathode is made from a piece of platinum foil or a bent platinum wire.

If a platinum dish is not available, one of glass can, of course, be used instead. In this case the specimen is placed on a piece of platinum foil, which is bent to an angle and connected to the positive supply as before.

When electrolytic etching is only employed occasionally it is possible to obtain satisfactory results without platinum electrodes, it only being necessary to ensure, as far as possible, that relatively insoluble electrodes are used as, otherwise, secondary reactions may have a disturbing effect on the result of the etching. For example, the specimen may be lowered into the electrolyte on a bent piece of lead sheet, provided with a lead wire to act both as a holder and as a conductor for the current, or, better still, held in a pair of nickel-chrome crucible tongs connected to the current supply. Graphite, in the form of arc-lamp carbon, makes a very good cathode, but, if necessary, a piece of sheet lead can be used.

As a general rule, in electrolytic etching, the specimen should be made electrically "alive" before lowering it into the etching reagent, because in this way a more even attack is obtained, and in some cases passivity is prevented.

The supply of current is best obtained from an accumulator with an E.M.F. of about 4 volts, but for occasional use an ordinary dry battery is generally sufficient. If direct current is available on the mains, the current can be tapped off potentiometrically (care being taken, of course, that this is done from the "earthed" side of the line). In most cases, however, electrolytic etching can be carried out quite successfully by coupling through the necessary series resistance (e.g. glow lamps) direct on to the D.C. mains, but then the holder for the "live" specimen must be provided with an insulated handle so that the specimen can be lowered into the electrolyte without danger of shock to the operator.

In permanent installations for electrolytic etching the electrical circuit should be provided with a fine regulating resistance and an ammeter for current strengths of 0·001–1 amp.

According to the A.S.T.M., electrolytic etching can be performed in an extremely simple way as follows.

The specimen, connected to the positive current supply, is set with the polished surface horizontal and upwards. On to the latter is poured as much of the electrolyte as it will retain, and into this is dipped the cathode, which is made of a bent piece of platinum wire. To obtain an even attack, the wire is moved about during the whole time of etching. The method is rather crude, but very easy, and usually succeeds quite well.

The following is another method of electrolytic etching which, according to the same authority, gives results considerably superior to the older processes in which the specimen is entirely immersed in the electrolyte.

The specimen, with the polished surface horizontal and downwards, is placed in an adjustable holder which is connected to the current supply. The vessel containing the electrolyte in which the cathode is immersed is placed under the specimen. The latter is now lowered just far enough definitely to wet the surface, and then immediately raised as high as the surface tension will permit without loss of contact with the electrolyte. A metal spring-clamp can be conveniently used as a holder for the specimen.

The solutions most commonly employed as electrolytes are 10 per cent ammonium chloride or sulphate, 0·5 per cent caustic soda, 1 per cent hydrochloric or sulphuric acid, etc.

According to Velguth nickel-chrome can be etched satisfactorily by electrolysis if it is first treated as the cathode in a 10 per cent solution of sodium hyposulphite, and immediately afterwards as the anode in 10 per cent hydrochloric acid. (See page 142.)

This method is probably applicable to a number of materials, such as iron-chromium alloys, which easily become passive.

Heat Tinting. When oxidation begins on a polished metal surface which is being heated, there appear the so-called "temper-colours" (heat-tints) which are due to the interference of light in the thin oxide film, and which, from their nature, correspond to the colours of Newton's rings.

The temper colours vary with the thickness of the oxide film.

According to Reiser the following are the corresponding temperatures and temper-colours for polished steel—

Pale yellow	220–230° C.	Violet	285°
Dark yellow	240°	Cornflower blue	295°
Yellowish brown	255°	Pale blue	315°
Brownish red	265°	Grey	330°
Purple red	275°		

On heating at a constant temperature, however, the length of time at that temperature is of great importance. Thus, at a tempering temperature of 230° C. a steel specimen shows the following colours—

After 15 min.	Pale yellow
,, 30 ,,	Red
,, 45 ,,	Blue
,, 60 ,,	Pale blue

If a material is composed of several structural constituents which have different susceptibilities to oxidation, they can be differentiated by tempering.

TABLE I

HEAT TINTS AT TEMPERATURES STEADILY RISING FROM 200° TO 400° C. (STEAD)

Pearlite 0·6% P, 0·75% C.	Ferrite	Cementite	Iron Phosphide	Manganese Sulphide
White	White	White	White, tinted yellow	Pale lavender blue
Very pale yellow	,,	,,	,,	,,
,,	,,	,,	,,	,,
Yellow	Very pale yellow	,,	,,	,,
,,	,,	,,	,,	,,
Yellow brown	Yellow	Very pale yellow	,,	,,
,,	,,	,,	,,	,,
Brown	Yellow brown	,,	,,	,,
,,	,,	,,	,,	,,
Purple red	Brown	Yellow	Very pale yellow	Pale yellow
,,	Purple red	Yellow brown	,,	,,
Blue	,,	,,	Pale yellow	,,
,,	Blue	Brown	Yellow	,,
Pale blue	,,	,,	Salmon	,,
,,	Pale blue	Red brown	Heliotrope	Brownish white
,,	,,	Purple red	Greenish	,,
Pale pea-green	,,	,,	Yellow	,,
,,	Pale pea-green	Blue	,,	,,
Pale yellow	Pale yellow	—	—	—
White	White	—	—	—

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TABLE II

HEAT TINTS AFTER DIFFERENT TIMES AT CONSTANT TEMPERATURE (STEAD)

(a) TEMPERING AT 232° C.

	Pearlite 0·6% P, 0·75% C.	Ferrite	Cementite	Iron Phosphide
1 min.	Very light yellow	White	Very light yellow	White
4 min.	Yellow brown	Very light yellow	Light yellow	,,
10 min.	,,	Darker yellow	,,	

(b) TEMPERING AT 280° C.

	Pearlite 0·6% P, 0·75% C.	Ferrite	Cementite	Iron Phosphide
1 min.	Brown	Yellow	Brownish yellow	White
2 min.	Purplish	Reddish brown		
3 min.	Dark purple	Brown		
4 min.	Blue	Purple	,,	Very light yellow
6 min.	,,		Orange brown	Light yellow
11 min.	,,	Blue	Red brown	Light salmon
15 min.	Light blue		Burnt sienna	,,
30 min.	Pale blue	Light blue	Purple brown	Heliotrope blue

(c) TEMPERING AT 326° C.

	Pearlite 0·6% P, 0·75% C.	Ferrite	Cementite	Iron Phosphide
1 min.	Purple red	Red	Yellow brown	Very pale yellow
2 min.	Dark blue	Purple red	Red brown	Pale yellow
3 min.	,,	,,	,,	,,
4 min.	,,	,,	,,	,,
5 min.	,,	Dark blue	,,	Pale salmon
6 min.	Pale blue	Pale blue	Purple red	Salmon
7 min.	Pale pea-green	Very pale blue	Purple	,,
8 min.	,,	,,	,,	,,
9 min.	Pale yellow	,,	,,	Purple
10 min.	,,	,,	,,	Heliotrope blue

The method appears to have been first used by Martens, and subsequently extended by Behrens, and Osmond, to alloys as well as steel, particularly copper alloys. Stead (2, 3) used heat-tinting for identifying iron phosphide as a structural constituent in steel. His observations are set out in Tables I and II.

Goerens and Dobbelstein observed that in a number of cases the thin oxide film adheres with different degrees of closeness to the metallic surface beneath. By careful repolishing after heat-tinting, therefore, certain constituents can be made bright so that greater contrast is obtained for photomicrography (*cf.* page 119).

Specimens which are to be photographed can, with advantage, be lightly etched before heat-tinting, so that the boundaries of the constituents are brought out more clearly.

For heat-tinting to be successful, it is essential for the polished surface to be absolutely clean and dry. It is, therefore, advisable to give the specimen a preliminary heating to about 120° C., followed by gentle rubbing on a warm piece of flannel before the real tempering, so that no moisture will subsequently condense on the surface.

Heat-tinting is most simply performed on a heated sheet or plate, but, in order to have a control over the temperature to some extent, the process can be carried out with the specimen floating in a bath of molten metal. Tin is very suitable for the latter. Stead (3) used a small electrically heated hot-plate, provided with an insulated support, which was placed on the stage of a portable microscope, so that the progress of the heat-tinting could be closely observed. If the process is used to a large extent as an etching method it is as well to fit up an air-oven with a thermostat.

When the desired temper-colour has been obtained, it is often recommended that the process be stopped by cooling the specimen in mercury or water, without, however, allowing the polished surface to come into contact with the liquid, otherwise stains will be produced. But it is, in general, more practical not to use a higher temperature than that at which 10 15 minutes are required to obtain the desired tint as, with this slow rate of etching, it is sufficient, with specimens of ordinary size at least, to place them on a cold metal support to stop the progress of the tempering.

An etching method closely related to heat-tinting consists of the formation of thin films on the polished metal surface by the use of bromine or iodine vapour, for example, or sulphuretted hydrogen. The process, however, has only a limited practical application.

Etching Under Fixed Surface Film, with Subsequent Colour Reaction. This method, due to Walldow and Benedicks, is described by the latter (3) as follows.

A 4 per cent solution of gelatine is heated to about 60° C. and spread in a thin layer over the surface of the slightly warmed specimen. After drying, the gelatine is hardened for ½–3 minutes in a 3 per cent solution of formalin. The specimen thus prepared is etched in a dilute acid, usually in alcoholic solution. After one or two minutes' etching it is washed in alcohol, and then treated with a solution of such a composition that characteristic colours are obtained with the products of the etching reaction which are held in the film.

According to Walldow* a layer of nitrocellulose may be used with advantage instead of gelatine. For this purpose colourless nitrocellulose lacquer is diluted with amyl acetate and applied to the surface of the specimen. After drying the etching procedure is as before.

Etching Under Fixed Surface Film, with Simultaneous Colour Reaction. This process is usually carried out with a solution of gelatine containing the etching reagent and the "indicator" together.

* Private communication.

The gelatine solution generally used is made up as follows: 5 grams of French gelatine are allowed to swell for half an hour in 20 c.c. of distilled water, and then dissolved by gentle heating on a water-bath. When solution is complete 10–20 c.c. of glycerine are added. This stock solution is rendered fluid for use by gently warming, and is then mixed with the appropriate chemicals dissolved in the least possible quantity of water. If chemicals, particularly acids, are added at an earlier stage the gelatine may possibly decompose.

The gelatine reagent is applied, with a glass rod or something similar, to the surface of the slightly warmed specimen, and is spread in a thin layer. If microscopic observations are to be made directly through the gelatine film it must be provided with a cover-glass. Under these circumstances it should be remembered that objectives with a focal distance shorter than about 16 mm. must be corrected for the cover-glass. When the latter is used one drop of the gelatine solution may be conveniently applied to the centre of the specimen, and the glass then laid upon it. The solution should be of such a consistency that the latter is pulled close to the surface of the specimen by capillary action. Care should be taken not to have too thick a film of gelatine between the specimen and the cover-glass.

In a number of cases the gelatine reagent, after etching for a suitable period, may be washed off with hot water, and then microscropic examination can be carried out in the usual way.

The method is similar to microchemical analysis, and is used for identifying sulphide inclusions in iron and steel (*cf.* page 120).

Etching Under a Removable Surface Film. This method makes use of a paper coated with a gelatine emulsion of an analytical reagent such as a silver or mercury salt, or potassium ferricyanide. The paper is soaked in a dilute solution of a suitable acid, and placed in contact with the surface of the specimen. The products formed as a result of the action of the acid react locally with the salts present in the surface of the paper, thereby producing characteristic colours.

After detaching from the specimen the paper shows the distribution of the substances which gave rise to the stains. The method is, of course, essentially macroscopic (*cf.* page 68).

A modification of this process has recently been published by Glazunov. In this the paper is soaked in a solution of an appropriate salt, e.g. potassium ferricyanide, without the addition of acid. The wet paper is then laid between the surface of the specimen and another flat metal surface. By means of electrolysis (specimen as anode) material is dissolved out from the specimen, thus bringing about a colour reaction in the paper.

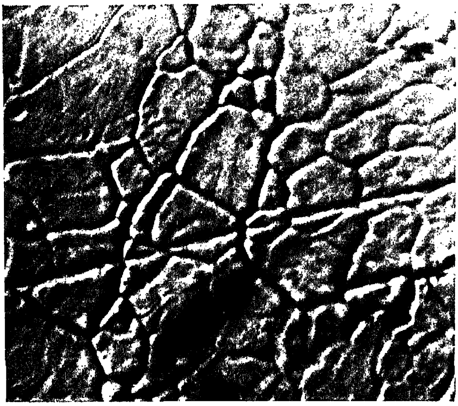

FIG. 4 GRAIN BOUNDARIES OF IRON ETCHED FOR 2 HOURS IN HYDROGEN AT 750° C. × 1,000
(Bureau of Standards)

Etching with Fused Salts. Saniter etched iron and steel at annealing temperatures by immersing the specimen in calcium chloride kept molten in a platinum crucible. This method of etching appears, however, to have been very uncertain in its results.

Etching at High Temperatures in Vacuo or in a Neutral Gas. After heating a polished specimen of iron or steel in a vacuum or a neutral atmosphere the grain boundaries can be seen upon it in the positions they occupied at the high temperature concerned.[1] The method has been used by a number of investigators, including Kroll, Rawdon and Scott, Hemingway and Ensminger, Oberhoffer and Heger, and Rawdon and Berglund, but is of minor importance in practice. Fig. 4 shows an example of this kind of etching.

Etching at High Temperatures with a Chemically Active Gas. Oberhoffer (1), Baykoff, Wark, and others have etched iron and steel by first heating the specimen to the desired temperature in vacuo or in a neutral gas, such as nitrogen, and then admitting a small quantity of hydrochloric acid. Like the previous method, this one is probably seldom used.

Etching by Cathodic Disintegration. Smith reports that certain alloys can be etched satisfactorily by cathodic disintegration. The specimen is made the cathode in a vacuum tube (0·05–0·005 mm. Hg) of which the anode is aluminium. The tube is operated at 2,000–7,500 volts with direct or alternating current for from 15 seconds to 10 minutes.

The behaviour of the alloys treated in this way is variable. A good etch is obtained in 15 seconds with copper-silver alloys, the copper-rich solid solution being coloured dark brown. In the cast alloys the primary and eutectic constituents appear particularly clearly. The criterion for good etching appears to be that the alloys must consist of two or more phases with marked differences in their ease of sputtering. Single phase alloys, accordingly, cannot be etched in this way.

A copper-zinc alloy containing 28 per cent Cu was well etched after ten minutes' treatment, but the eutectic alloys of cadmium with zinc, lead, or tin could not be etched by this method.

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[1] According to a private communication from B. Kjerrman, the etching effect is probably due to gases evolved by the steel.

CHAPTER III

MACRO-ETCHING OF IRON AND STEEL.

Introduction. The macro-etching of iron and steel, whereby the more pronounced differences of a chemical or physical nature in the material are brought to light, is one of the most valuable aids for discovering structural defects. Quite often such etching shows, to a certain extent, the history of the material right from the ingot to the finished product. It is of special value for ascertaining from the appearance of the so-called "fibre structure" the mode of flow of the material in hammered or pressed parts. At the same time, however, the interpretation of the result of macro-etching demands a certain amount of discrimination.

The etching reagents used are acids, iodine solutions, ammonium persulphate solution, and neutral or acid solutions containing copper. Acids are used to a large extent owing to their simple manipulation and cheapness, the latter being an important point when the control of material on a large scale is concerned. They give a good general survey of the condition of the material. Iodine solutions have been used in the past very considerably, but nowadays they are employed in exceptional cases only. Ammonium persulphate has its own particular use for etching the surfaces of the crystal grains, and for the examination of welded joints and seams. Reagents containing copper are specially employed for etching up any possible dendritic structure, for indicating phosphorus segregation, and for investigating suspected cold-working.

Preparation of the Specimen for Macro-etching. The following may be added to what has already been stated in the introductory chapter regarding the preparation of specimens in general (see Johnson, Keshian, Bureau of Standards, and others).

When preparing a sample ready for macroscopic work the condition of the test-surface should be directly related to the action of the etching reagent and the object of the investigation. Thus, to carry out a prolonged deep-etching with strong acid on a finely ground specimen is a waste of the time and effort expended on the latter because a planed or turned surface is usually quite sufficient in such a case. On the other hand, a number of the reagents containing copper require a carefully ground and polished surface to give the best result. As a general rule, the more superficial the etching, the more finished must be the preparation of the specimen. For example, when the chief object is a study of crystal grain size and shape a relatively light etching only is employed, so that the surface of the specimen must be carefully polished.

When examining the surface of hardened steel for soft spots it is only necessary to grind off carefully any existing annealing scale so that a clean metallic surface is obtained.

There are, however, many cases where hardly any working-up of the specimen is needed. Thus, for example, to detect the presence of pipe in a heavy, rolled billet, deep etching direct on a hot-sheared section is sufficient. For major segregations and pipe in lighter materials it is often feasible to use a section as cut in the cold. For discovering surface defects due to rolling, etc., a convenient length of the material may be etched without any special preparation. This results in the dissolving out of any scale which may have been rolled into the surface, together with any thin fins of metal which may have been formed into seams. But if it is intended to make a more careful examination of the material for segregations, cracks, dendritic structure, etc., then a section must be cut and planed or turned.

Under certain circumstances it may require grinding and, more rarely, polishing.

When examining round, rolled material it is often advantageous to reduce the diameter in a stair-like formation by turning a suitable number of steps on a lathe; the last cut for each diameter is made so as to obtain the smoothest possible surface (Johnson). Any fibre structure and slag threads can, in this way, be more easily studied than in an ordinary longitudinal section. After a little practice, excess aluminium oxide, and slag in general, can be easily observed in the unetched fine-cut surface owing to their different reflecting powers. They appear as long or short lines parallel to the direction of rolling.

Cleaning the Surface Before Etching. Having completed the preparation of the specimen, the surface must be suitably cleaned free from grease. Ast (see Frémont) recommends a solution of sodium- or potassium-hydroxide in alcohol for this purpose. Alternatively, immediately before etching, the surface can be rubbed over with emery paper, or with an otherwise clean rag dipped in a mixture of emery powder and spirit, or of French chalk and water, etc. As soon as the etching reagent begins its action, a watch is kept for the appearance of any stains which might be due to residual grease; if these appear the specimen is taken out, washed, and again rubbed over with the emery paper or cleaning mixture and the etching then continued.

A Note on Protective Measures. When deep-etching with acids, particularly hot acids, means must generally be provided for removing the evolved gases, since these are formed in considerable quantities, and are unhealthy, not only in themselves, but also because of the extremely small drops of liquid which are thrown out when the bubbles of gas leave the surface.

Also, it is often advisable to use rubber gloves when handling specimens for macro-etching.

Macro-etching with Acids. Etching with acids brings out inequalities in the composition as well as in the physical condition of the material, so that the result can, therefore, be said to be of a general orientating character. The effect, depending as it does on the strength of the reagent, and on the time of etching, can be extremely variable for the same material. According to Rawdon (2) it is a general rule that strong acids, which etch in a short time, give better contrasts than dilute, which need a longer period.

The acids which are chiefly concerned are picric, nitric, sulphuric, or hydrochloric. They are used individually in a wide variety of concentrations in alcohol or water, and together in a large number of different mixtures.

Picric Acid. A 3 per cent solution of picric acid in ethyl alcohol is frequently used for showing segregations, etc. The etching is carried out at the ordinary temperature for a period of four to five hours.

"Hütte" recommends a solution of 1 gram picric acid in 25 c.c. alcohol and 1 drop conc. HCl, this solution being used hot.

Stead (5) advocates Le Chatelier's method of etching with a 0·5 per cent aqueous solution of picric acid for bringing out unequal distribution of phosphorus. The areas which are stained soonest have the highest phosphorus content.

Nitric Acid. A 10–15 per cent solution of nitric acid in water or alcohol is generally employed. According to Keshian it is very suitable for indicating segregations in general. For small local segregations, however, nitric acid is of less value. The contrasts are relatively weak, and no marked relief effect obtained.

Leitner successfully used a 5 per cent aqueous or alcoholic solution for etching nickel-chrome steel containing approximately 0·10 per cent C, 0·20 per cent Si, 0·30 per cent Mn, 4 per cent Ni, and 0·90 per cent

Cr. The time of etching was varied somewhat according to the composition of the material, but, as a rule, did not exceed half an hour. During etching the surface of the specimen becomes coated after a few minutes with a brown layer through which dendrites, if any, may subsequently be observed. After a certain period the layer becomes black, and then the etching is considered finished.

In a similar steel, but with a carbon content of 0·30-0·40 per cent, it was not possible to obtain a contrast sufficiently strong for photography. If, however, the specimen, after cleaning, was re-etched for a short time, the grain boundaries were darkened.

The Iron and Steel Institute's Heterogeneity Research Committee, in a report on ingot defects, recommends etching with nitric acid when dealing with large surfaces, such as the entire cross-sections of ingots, for example, because it is not always possible to prepare them very well. To remove machining marks most efficiently the etching is commenced with 2 per cent acid, then the concentration is raised gradually to between 5 and 8 per cent, and the etching finally completed with 10 per cent acid. In this way a moderately deep etch is obtained in a reasonable time, and with the greatest elimination of scratches.

The A.S.T.M. (1) recommends a mixture of 25 per cent nitric acid (s.g. 1·92) and 75 per cent water for the general development of structure.

Sulphuric Acid. According to Keshian a hot 10-20 per cent aqueous solution is most suitable. It appears to attack selectively the sulphide inclusions in the steel, but, as regards the macro-structure in general, it gives results inferior to the other acids or mixtures of them.

For the macroscopic investigation of large specimens, such as cross-sections from billets about 100 mm. square, 10 per cent sulphuric acid in water is used,[1] and the sections are allowed to remain in this solution for about 24 hours. Nearly the same effect is obtained in 6 hours with a 20 per cent solution. The sections do not need to be polished, but only levelled in a lathe or planing machine.

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[1] Communication from a Swedish works.

With this method the result obtained corresponds to the Baumann print on silver bromide paper, and, in fact, where it is used, it is considered to be superior to the latter process, since piping, blowholes, and porosity are also brought out, and, in certain cases, even the primary structure.

Hydrochloric Acid. Keshian states that a hot solution of 1 part conc. HCl and 1 part water is very often used. The specimen is placed in a porcelain dish with the ground surface upwards, and the solution poured in so that the section is entirely covered. The dish is placed on a hot-plate, and the reagent kept just under boiling-point for half to one hour. The specimen is taken out by means of tongs, washed in hot water, and dried with alcohol. With hydrochloric acid, heterogeneities in general are brought out very well.

The undiluted acid is also used to a great extent at a temperature of about 100° C. The time of etching is from 10 minutes to half an hour or longer. Harper reports obtaining useful information on nickel steels containing 3–3·5 per cent Ni with this method when a standard size of specimen, and the same etching procedure, were used throughout.

For discovering soft spots in the surface of hardened steel, the latter, carefully ground clean, is etched for about 5 seconds in cold 50 per cent hydrochloric acid, whereupon the soft spots, if any, appear dark coloured.

Mixtures of Various Acids. Numerous mixtures of hydrochloric and sulphuric acids are used for deep etching, and they are considered by many to give results considerably better than either of the two acids alone.

Sauveur, according to the A.S.S.T. Handbook, recommends a mixture of 3 parts water, 2 parts conc. sulphuric acid, and 1 part conc. hydrochloric. When making up the reagent the sulphuric acid is mixed with the water first and then, after cooling somewhat, the hydrochloric is added. The solution is used at a temperature near the boiling point. The etching period is at least half an hour, or until the defects, if any, appear. It is used for general development of the structure.

The A.S.T.M. (1) recommends the following mixture: 10 vols. conc. HCl, 30 vols. conc. H_2SO_4, and 60 vols. water.

In certain places in Germany a suitable pickling inhibitor is added to these acid mixtures, with the result that a more even attack is obtained, particularly in the case of nickel-chrome steels.

Waring and Hofammann used a combination of 9 parts HCl, 3 parts H_2SO_4, and 1 part water, for etching rail sections. The etching time was about two hours in the nearly boiling solution.

Keshian recommends as an excellent universal solution for deep etching: Hydrochloric acid 500 c.c. Sulphuric acid 70 c.c. Water 180 c.c. When etching in the nearly boiling solution, alloy steels and high carbon steels require a period of 1–2 hours, and softer metals ½–1 hour.

Interpretation of the Etched Surface (H. G. Keshian). In macroscopic deep-etching with acids a wide variety of results can be obtained from the different steels. The specimen may show evidence of seams, cracks, piping, or pinholes; it may have a dense or porous appearance, be smooth or rough on the surface, and have a light or dark colour; the structure may to a greater or less degree be dendritic; segregations may appear as strongly attacked areas. The question then arises as to how the etched surface is to be interpreted; what indicates a good steel, and what a bad?

As soon as the material exhibits the more serious defects, such as cracks, piping, large slag inclusions, pronounced segregations, etc., the material is, of course, classed as inferior. Beyond this, however, it is a difficult matter to say anything, and the interpretation calls for great experience, because a large number of the differences in the result of etching can be ascribed to the chemical analysis of the material, and its previous mechanical working and heat-treatment. Due consideration must be given to these circumstances.

Effect of the Composition of the Material. Steels with a carbon content of up to about 0·50 per cent dissolve more rapidly than those with higher carbon.[1] For this reason they appear rougher after etching, and less dense, and have a darker colour. Alloy steels containing about 1½ per cent tungsten etch darker, and more uniformly, than ordinary carbon steels, and have a closer texture. The nickel-chrome steels, and oil-hardening manganese steels, acquire an appearance which is intermediate to the high alloy steels and the high carbon steels. High-speed steel always etches very dark, and presents a structure which appears the densest and smoothest of all. Next in order come the heat-resisting chromium steels. Steels containing vanadium appear somewhat more dense than the corresponding steels without vanadium, but are very little different as regards colour. Wrought-iron is recognized by its pronounced fibre-structure. On the other hand, ordinary malleable ingot-iron can hardly be distinguished from a 0·50 per cent carbon steel, for example.

Effect of the Quality of the Material. Crucible and electric steels etch with a closer texture, and more evenly, than acid open-hearth steels. The latter etch somewhat darker and acquire a granular appearance. The former are difficult to differentiate by deep-etching.

[1] According to Endô this is not the case, the rate of solution, on the contrary, rising approximately proportionally with the carbon content up to the eutectoid composition.

Effect of Mechanical Working. The mechanical working of the material has a marked influence on the result of etching. The more thorough the working, the more even and smooth is the effect in the etched cross-section generally. This can easily be proved if an originally dendritic billet is rolled down to various smaller dimensions. In this respect, however, exceptions will occur in the case of those materials which have strongly marked fibre-structures. Transverse sections of such materials always have a coarse structure, even if they are satisfactorily worked. These conditions often occur if a dendritic material is rolled down with heavy reductions.

Effect of the Direction of the Fibre. When etching a section at right angles to the direction of the fibres a greater rate of solution, and hence a coarser appearance, is obtained than when etching a section parallel to them. Transverse sections are, therefore, always darker and more porous, than longitudinal.

Influence of the Temperature of the Etching Solution. The temperature of the etching reagent has a considerable influence on the appearance of the etched section. At higher temperatures the rate of solution is greater, and the material gives a coarser effect. The dependence of the rate of solution on the temperature is illustrated in the Table on page 45. The specimens consisted of cylinders $\frac{3}{4}$ in. in diameter, by 1 in. long.

Effect of Time of Etching. The length of the etching period exerts a marked influence on the result. The longer the etching, the coarser is the appearance of the surface. The time of etching must, therefore, always be kept constant in comparative tests. It should be remembered, however, that even a material free from defects shows, over its entire surface, a roughness which increases uniformly as the etching is prolonged.

Effect of Heat Treatment. Hardened steel has, after etching, a dense and smooth surface. A hardened and

TABLE III

LOSS IN WEIGHT PER SQ. CM. AFTER TWO HOURS ETCHING IN A MIXTURE OF 500 C.C. HCl, 70 C.C. H_2SO_4, AND 200 C.C. WATER, AT THE TEMPERATURES STATED

Analysis of the Steel							Loss in weight, grams per cm.² when etched at		
C	Mn	Si	Cr		W	V	38°	65°	94° C.
0·19	0·76	0·14					0·164	0·582	1·369
1·12	0·36	0·10					0·150	0·396	0·803
0·40	1·36	0·18					0·333	1·093	1·757
0·65					17·05	0·79	0·107	0·411	0·771
0·16				3·54			0·097	0·300	0·631
2·05			11·42				0·368	0·705	1·408
1·22			0·50		1·51		0·111	0·300	0·594
0·34			13·58				0·300	0·980	1·871
1·30			0·24		3·74		0·145	0·424	0·821

tempered material etches quite differently from one merely hardened. The effect of tempering becomes noticeable from 150° C. up to about 400° C. Further tempering at up to 650° C. does not bring about any greater change in respect to the etching. As soon, however, as the material is fully annealed the etched structure becomes coarser. Hardened steel cracks very easily on deep-etching, particularly in hot acids. This may frequently occur even after tempering at 370° C. In general, therefore, hardened steel should be adequately annealed before it is deeply etched.

Various Macro-structures and Their Significance as Indicators of the Properties of the Material. Five main types can be distinguished—

1. The material exhibits obvious defects in the form of actual discontinuities in the metal.

Such defects as cracks, piping, seams, blowholes, slag inclusions, and the presence of unmelted steel scrap speak for themselves, and if deep-etching is used as a control test, material containing them need never pass into service.

2. The material exhibits distinct segregations.

Segregations appear in the etched surface as dark patches of various sizes. The nature of the segregation cannot be deduced from the appearance of the etching, so other methods of investigation, such as chemical analysis or microscopic examination, must also be employed. Large segregates of a serious nature are, of course, the sign of an inferior material. On the other hand, in those cases where, according to the etching test, only moderate segregates occur, it is extremely difficult to decide the real degree of segregation, and, therefore, great caution should be observed as regards the question of rejection merely on the basis of the etching result.

3. The material has a smooth and even surface after etching.

In general this indicates a good and uniform structure in the material, though sometimes such etching results can be misleading. If, however, two specimens are of similar material, and have been subjected to the same mechanical working and heat treatment, but one of them after etching has a denser and smoother appearance, it is extremely probable that it is better than the material with the coarser surface. In a case like this, however, the result of etching is obviously not a test giving absolute proof of quality.

4. The material has a porous appearance after etching.

This points to a certain degree of chemical heterogeneity in the material, and it occurs readily in the central parts of the section. In such cases only experience can decide as to the suitability of the material for its purpose. The method of rolling has a great deal to do with the appearance of this etching structure.

5. The material exhibits a dendritic structure.

Steels with high carbon content seem to be more prone to acquire a dentritic structure than those with low carbon. Alloy steels, though often with the exception of the nickel-chrome steels, are better in this respect than carbon steels, while high-speed steels very seldom show it. Abnormally well-defined dendritic structure is, of course, objectionable; as regards the normal type, however, no general statement can be made since in many cases it plays a very minor part, while in others it is important. A material may seem quite free from the structure in a section perpendicular to the direction of rolling, and yet show it in a longitudinal section.

Macro-etching with Iodine Solution. Frémont states that iodine was introduced as an etching reagent for iron and steel in 1895 by Osmond in the form of "tincture of iodine" (iodine dissolved in ethyl alcohol). Reinhardt replaced the alcohol as the solvent by an aqueous solution of potassium iodide. Tetmajor considered the most suitable proportions to be 1 part iodine in 2 parts of iodide and 10 parts water.

Frémont recommends iodine solution for showing pipe formations, and is of the opinion that, in macroscopic work, this reagent gives etching effects which, in general, are more suitable for photography than those which are obtained with acids, ferric chloride, mercuric chloride, or copper-ammonium chloride, while for ocular observation the effect is very much the same. According to the Bureau of Standards (2), however, copper-ammonium chloride gives a more clearly defined result.

Iodine was formerly used quite extensively, but nowadays it is probably seldom employed.

According to a report by Oberhoffer (6) a solution of 1 part decinormal iodine solution in 10 parts alcohol should act as an indicator of the distribution of silicon in steel. However, there is no further mention of this observation after the first short communication, and therefore the matter seems to be rather in doubt.

Macro-etching with Ammonium Persulphate. Ammonium persulphate seems to have been first proposed as an etching reagent for iron and steel by Czochralski (1) in 1915, but it was not used very much before about 1920, when Rawdon (1) published a number of articles on its great merits for macro-etching. The following remarks are based mainly on his work.

Ammonium persulphate is a powerful oxidizing agent. The chief reaction during etching is

$$2\,Fe + 3\,S_2O_8'' \rightarrow 2\,Fe''' + 6\,SO_4''$$

The persulphate ion, however, slowly breaks down in water and oxygen is evolved, thus

$$2\,S_2O_8'' + 2H_2O \rightarrow 4H' + 4\,SO_4'' + O_2$$

In consequence of this the solution should only be made up just before use.

A 10 per cent solution in water has been found to give good results, but the concentration can be raised to 20 per cent without inconvenience. The etching is carried out best by "swabbing." For a well prepared surface an etching period of 1–2 minutes is generally sufficient. In this case the grinding of the section is usually completed with emery paper. The etching of large sections can be done quite well direct on surfaces planed smooth, but the time of etching must then be extended to half an hour, or possibly somewhat longer. Under these circumstances it is an advantage to warm the etching solution to 50–60° C. When the etching period is so prolonged the specimen may be conveniently immersed entirely in the solution, and the surface rubbed with a swab of cotton wool wrapped firmly round a wooden stick. If the solution shows a tendency to weaken in its etching action some more salt is added. As soon as the desired etching effect is obtained the section is washed in water, and treated subsequently in the usual way.

Photography of the surface is done best with the specimen under water, alcohol, or thin oil. If this procedure is inconvenient owing to the size of the specimen, or other circumstances, a result almost as good may be obtained by painting the surface with glycerine or oil, or by varnishing.[1] It may be mentioned that these remarks hold good for all photography of macro-etched surfaces when the greatest possible contrast is desired.

Ammonium persulphate is the best reagent existing for the macroscopic grain-contrast etching of iron and steel. Hence it may be used with advantage to show recrystallization phenomena. For investigations on welded joints of various kinds it is often unsurpassed (Rawdon (I), Jimeno and del Fresno, and several others), although, for completing the information obtained, etching with a second reagent may also be advisable. It is very suitable for etching such materials as wrought iron, which, as a rule, is characterized by uneven grain-size. One great advantage in ammonium persulphate etching is that the attack is, in reality, very superficial, so that, if desired, the surface can easily be ground clean again for a subsequent microscopical examination. This is often impossible after deep-etching with acids without removing considerable quantities of metal.

Ammonium persulphate also brings out clearly any variations in chemical composition, such as segregations, etc. In this respect the effect is somewhat similar to that obtained with other macroscopic etching reagents. The fibre-structure can sometimes be etched up better with ammonium persulphate than with other solutions.

Another advantage of this reagent lies in its slight effect on the skin in comparison with acids or copper reagents, and hence its manipulation is simpler.

Macro-etching with Reagents Containing Copper. These etching reagents are particularly sensitive to differences in the distributions of phosphorus and carbon. At the same time, some of them produce a slight grain-contrast etch, and, for these reasons, are the best reagents for showing dendritic structure (primary etching). According to several investigators the result of etching is also dependent on the occurrence of dissolved oxygen in the iron, but this is disputed by others (see page 56).

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[1] The commercial metal varnishes (lacquers) should be diluted with about 2–3 times their volume of a suitable reducer.

Sometimes the copper-containing macro-etching reagents are also used for etching for microscopic purposes, but in such cases they are generally diluted.

A list comparing the solutions described below is given in Table IV on page 66.

Neutral Copper-ammonium Chloride. The first, and for long most used, of the copper-containing etching reagents, is the one suggested by Heyn in 1906, consisting of a solution of copper-ammonium chloride in water in the proportion of 1 : 12. This concentration is chosen so that the precipitated copper layer is porous and spongy, and, therefore, can be easily washed off at the conclusion of etching.

The etching itself is carried out with the specimen completely immersed in the solution, which is contained in a dish. Care must be taken that the entire surface is quickly and evenly covered by the reagent, and that no air bubbles form and remain attached to it. Before being placed in the solution, the specimen should be thoroughly cleaned with alcohol or whiting, for example. During etching the dish is kept in motion so that any differences of concentration which may arise in the solution, are neutralized as soon as possible. For finely ground surfaces a suitable etching period is, in general, about one minute; rougher surfaces require a longer time. After etching the specimen is washed in water and the precipitated copper cleaned off with cotton wool or some similar material. If the copper should show a tendency to stick in certain places, it is often useful to rub the surface with a wet cork. If this is no help, the copper can be removed by treatment with either strong ammonia, or ammoniacal copper-ammonium chloride, or a 0·5 per cent solution of ammonium persulphate. When the copper layer has been removed, the specimen is washed and dried in the usual way.

During etching with copper-ammonium chloride the areas high in carbon or phosphorus are attacked more strongly than the purer parts, and thus appear darker than their surroundings when viewed in diffuse illumination. If the phosphorus content is very high a slight brown colouration may also appear—due presumably to the superficial formation of phosphor-copper. Locally cold-worked areas, such as the surroundings of a rivet-hole, are also attacked strongly by the reagent.

This method of etching is used preferably for iron and mild steel with a carbon content under 0·3 per cent, but nevertheless it can also be employed for steels containing up to 0·7 per cent carbon. With higher carbon contents, however, the difficulties of removing the copper layer become excessive. In such cases the etching reagent proposed by Oberhoffer (2) may be used as an alternative (see page 57). The method is not suitable for graphitic cast irons, hardened steels, or high alloy steels.

According to Haufe this method is, for soft iron at least, just as good as etching with more complex solutions.

The Bureau of Standards recommends a solution somewhat more dilute than that of Heyn. Also, in this modified solution, the section is etched a number of times with intermediate washings. According to a report from a Swedish works, this method has been found to be quite satisfactory.

In certain cases it is possible to start with a planed surface, and etch it at room temperature with a saturated solution of technically pure copper-ammonium chloride.[1] In this way dendritic structures, segregates, flakes, etc., are brought out (see page 77). The specimen is immersed in the solution with the smooth planed surface upwards, and allowed to remain, say, overnight, then taken out and brushed clean in water. It is finally washed with soda solution, for example, to inhibit the formation of rust. For this method of etching to be successful the steel should have a moderately fine structure.

The method is advantageous in practice because the laborious grinding and polishing of the specimen is avoided; it is, therefore, easy to do, and requires little supervision.

Copper Sulphate Solution. Belynski used a 1 per cent solution of copper sulphate to show up dendrites, etc., in irons and steels with carbon contents of 0·1–1·3 per cent. This reagent has not been used very much.

Acid Copper-ammonium Chloride. Humfrey used a solution of copper-ammonium chloride, acidified with hydrochloric acid, for macro-etching large surfaces. With this reagent a very strong and sharp relief effect was obtained, with the result that the etched surfaces could be used as "clichés" for taking off prints on to paper by means of printers' ink. The following was the composition of the solution.

Copper ammonium chloride	120 grams
Hydrochloric acid, conc. .	50 c.c.
Water	1,000 c.c.

Occasionally, as for nickel- and chromium-alloy steels, the best etch was obtained if the reagent was made slightly less acid.

From this acid solution, however, the copper is precipitated on the specimen as a strongly adherent layer, and, therefore, the etching must always be commenced in the neutral solution. With the latter the process is continued until it is certain that all scratches or machining marks have been removed; the solution is then made more acid with successive additions of HCl, without, however, removing the layer of copper already deposited. The period of etching varies from 20 minutes to 1–2 hours. If deep marks are left on the surface of the section from the previous machining, it is often advantageous to etch first with dilute nitric acid till these scratches have disappeared; subsequently the process is continued, first with the neutral, and finally with the acid solution.

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[1] Communication from a Swedish works.

When the etching is finished the copper is washed off, as after using Heyn's neutral solution. If a deeper etching than the one obtained is desired the process is continued, but it is necessary to start again in the neutral solution. Greater contrasts are obtained if the section is polished slightly with fine emery-paper after etching.

For etching ingot sections the Iron and Steel Institute's Heterogeneity Research Committee recommends the following procedure. Etch first in an 8–12 per cent neutral solution for 10 minutes to half-an-hour. Then for half an hour with a solution to which has been added 2·5–5 per cent HCl. Continue for a third half-hour with an addition of 15 per cent HCl. An intermediate etching with 7·5–10 per cent of HCl present may also be carried out. If necessary, more strongly acid solutions can be used to produce an adequate relief-effect.

Allison and Rock used Humfrey's method of etching in order to bring out any possible dendritic structure in steel castings[1] containing 0·07 per cent C, 0·06 per cent Si, 0·05 per cent Mn, and about 0·06 per cent P.

Whiteley (2) employed the following solution—

Ferric chloride	200 grams.
Cupric chloride	1·2 grams.
Hydrochloric acid	100 c.c.
Water	1,000 c.c.

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[1] For use in the electrical industry.

After five minutes' etching the specimen was taken out, the surface ground with fine emery paper, and the etching then continued for half an hour or more. With this method the relief-effect is not quite so sharp as with Humfrey's process.

Whiteley's solution is used in a Swedish works, with the exception that only 1·0 gram of copper chloride is taken. The etching time is 20 minutes. It has been found convenient to take the specimen out after 2 minutes, polish it with No. 00 emery paper, and then continue the etching.

According to the A.S.S.T. Handbook, Dickenson has proposed a reagent of the composition—

Ferric chloride .	40 grams.
Cupric chloride .	3 grams.
Hydrochloric acid	40 c.c.
Water . .	500 c.c.

Before treatment in this solution the specimen is etched with 10 per cent nitric acid.

Rosenhain and Haughton introduced another type of copper-reagent, which, besides the copper salt, contains hydrochloric acid, ferric chloride, etc., in such proportions that copper is precipitated as a strongly adherent, but thin, layer on certain areas of the surface of the specimen, while the others remain as clear as before etching.

The following is the composition of this solution—

Ferric chloride	30 grams.
Hydrochloric acid, conc.	100 c.c.
Cupric chloride	1 gram.
Stannous chloride	0·5 gram.
Water	1,000 c.c.

The time of etching is 10 secs. to 2 minutes, and the solution should not be used more than once. The specimen which is to undergo treatment should be polished as for ordinary microscopic etching. In pearlitic steels which have a uniform distribution of phosphorus, the copper is deposited on the ferrite, causing the latter to appear dark, while the pearlite is unchanged. When the phosphorus is unevenly distributed the areas wherein it is highest are left clear. This etching reagent is more adapted to microscopic investigation than macroscopic. At the same time, however, very deep pitting occurs round any small non-metallic inclusions which may be present in the surface. To avoid this the A.S.S.T. Handbook recommends dilution of the solution. A certain amount of experience is necessary to obtain a satisfactory etch, and the specimen must be perfectly clean.

Stead's Etching Reagents. Stead (4) improved the above reagent by the use of alcohol, and suggested the following composition—

Cupric chloride	10 grams.	Stead's No. 1 reagent.
Magnesium chloride	40 grams.	
Hydrochloric acid	20 c.c.	
Ethyl alcohol up to	1,000 c.c.	

The salts are first dissolved in the minimum quantity of hot water, amounting to approximately 10 per cent of their weight, and then the alcohol is added. The etching is carried out by allowing a few drops of the solution to remain on the surface of the specimen for about one minute, then pouring them off, and replacing them with fresh solution as often as required. When sufficiently etched the specimen is washed with hot water and alcohol. The copper deposits first as a strongly adherent layer on the purest iron, and then, as the etching is continued, spreads out gradually over the less pure parts as well. Areas with extremely high phosphorus contents, however, remain without a covering of copper even after very long etching.

Haufe[1] tries to explain the effect on the assumption that the copper is deposited first as an extremely thin film on the least pure areas, and protects these from further attack. The same investigator considers that

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[1] Haufe mentions water instead of alcohol (misprint?).

Stead's reagent is not very suitable for showing phosphorous segregations, because the details are indistinct.

Le Chatelier and Bogitch consider that the amount of oxygen dissolved in the steel is a deciding factor in the etching effect. Thus they state that the strongest effect is obtained with an oxygen content of 0·01 per cent, and that with lower or higher concentrations it decreases, while if the oxygen content is very low, Stead's reagent does not etch at all. Tritton and Hanson, however, do not agree that the oxygen content has such an influence on the result.

Stead (5) recommends, besides the foregoing, the use of a stronger solution.

Cupric chloride .	5 grams.	. Stead's No. 2 reagent.
Magnesium chloride	4 grams.	
Hydrochloric acid	1 c.c.	
Water . .	20 c.c.	
Alcohol . .	100 c.c.	

The ordinary, dilute, solution No. 1 is used for low percentages of phosphorus, while the stronger No. 2 is used for high percentages. Etching time about 1 minute.

Comstock (2) used a somewhat modified composition of Stead's reagent for investigating transverse fissures in rails.

Cupric chloride .	2·5 grams.	The salts are dissolved in the hydrochloric acid, with the addition of the least possible quantity of hot water.
Magnesium chloride	10 grams.	
Hydrochloric acid	5 c.c.	
Alcohol up to .	250 c.c.	

If the etching with this solution was not too prolonged, the phosphorus-rich areas could be kept clear. According to Charpy and Bonnerot the contrasts can, under certain circumstances, be increased by dissolving off the precipitated copper by rubbing with strong ammonia. This latter process is also recommended by Comstock when examining, at magnifications of 20–50 the dendritic structure brought out by Stead's ordinary reagent. In this case the deposited copper layer, which will subsequently be removed, is allowed to become relatively thick.

Le Chatelier and Lemoine's etching reagent, often called Le Chatelier's No. 1 reagent, is only a slight modification of Stead's No. 1 solution. The composition is—

Cupric chloride	1 gram.
Magnesium chloride	4 grams.
Hydrochloric acid, conc.	2 c.c.
Water	18 c.c.
Methyl or ethyl alcohol	100 c.c.

(It is important for the alcohol to be absolute in order that the total amount of water present may be no greater than strictly necessary.)

This solution is used similarly to Stead's reagent, except that Le Chatelier carries out the etching by immersing the specimen in the solution. The etching contrasts can be increased by electrolysis, with the specimen as the anode; the current strength (for an ordinary specimen) should be less than 50 milliamps. A dry battery can be used to supply the current; (electrode distance about 2 cms.).

Le Chatelier and Dupuy's etching reagent, often called Le Chatelier's No. 2 reagent, is—

Cupric chloride	1 gram.
Picric acid	0·5 gram.
Hydrochloric acid	1·3–2·5 c.c.
Water	10 c.c.
Ethyl alcohol	100 c.c.

The volume of HCl is varied according to the analysis of the specimen. Haufe reports that this solution is also suitable for electrolytic work, but it is said to be very capricious.

According to Hatfield, heterogeneous distribution of manganese and silicon can be shown equally well with Stead's reagent, or Le Chatelier's.

Oberhoffer's Reagent. After a preliminary study, Oberhoffer (2) put forward the following—which is considered to be an improvement as regards the obtaining of uniform results.

Cupric chloride	1 gram.
Stannous chloride	0·5 gram.
Ferric chloride	30 grams.
Hydrochloric acid,	30 c.c.
Water, distilled	500 c.c.
Ethyl alcohol.	500 c.c.

The specimen must be well ground and carefully polished, but according to some information received, instead of being etched immediately after polishing, it should first remain in the air for half to one hour. It is, furthermore, important for the specimen to be absolutely dry before etching, and also to be at the same temperature as the etching solution (Haufe).

Oberhoffer's solution acts somewhat more slowly than the previous reagents, and also more uniformly, with consequent less risk of staining. The etching is done normally, and the precipitation of copper is not noteworthy because the composition of the solution is so chosen that the deposition of copper on pure ferrite is just prevented.

The areas which have electrochemically less "noble" properties, e.g. ferrite containing phosphorus, acquire over their surfaces an extremely thin film of copper which protects them from further attack, so that they finally stand in relief because the purest, unprotected areas, are dissolved out by the hydrochloric acid and ferric chloride. This state of affairs is thus the direct opposite to that obtained with Heyn's, and similar, etching reagents.

The contrasts obtained may be further emphasized by repeated etchings, with light intermediate polishings, especially if the structure is fairly coarse.[1] Difficulties have, at times, been encountered when etching specimens soldered to another material.

To bring out dendrites and grain boundaries at the same time, Leitner recommends a double-etching. The first etch is with 5 per cent nitric acid in alcohol or water, and the second with Oberhoffer's reagent.

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[1] Pointed out by a Swedish works.

For micro-etching, 1 part of the macro-reagent is added to 10 parts of alcohol. Haufe considers, however, that, with Oberhoffer's reagent, the surface is roughened too much for microscopic work.

Fry's Etching Reagents. Fry (2), who undertook a further critical investigation of the etching reagents containing copper, showed that, for plain carbon steels, stannous chloride is unnecessary, and that the concentration of alcohol can be increased, a point which is of special advantage in micro-etching. Similarly it was found that a cleaner structure was obtained by omitting the ferric chloride. A solution of the following composition appeared to be specially useful.

Cupric chloride . . .	1·5 gram.	Fry's No. 1 etching reagent.
Hydrochloric acid . .	30 c.c.	
Water	95 c.c.	
Ethyl alcohol . . .	30 c.c.	

Particularly good grain-contrast etchings are obtained with this solution. Very careful polishing of the specimen is, of course, necessary.

When etching high alloy steels it often happens that Oberhoffer's reagent proves to be quite useless, because copper is deposited over the entire surface of the specimen. In such cases useful results can often be obtained by increasing the proportion of hydrochloric acid, or by using Fry's No. 1 reagent, or the following—

Cupric chloride . . .	4 grams	Fry's No. 2 etching reagent.
Hydrochloric acid, conc. .	20 c.c.	
Water	40 c.c.	
Ethyl alcohol . . .	20 c.c.	

Fry's reagents, Nos. 1 and 2, can be used quite well for showing the degree of segregation of the phosphorus, because solution No. 2 only leaves unattacked the areas containing high phosphorus, while the No. 1

solution similarly leaves the areas of moderate phosphorus content. The two solutions can, with advantage, be mixed in various proportions.

Haufe considers that no essential difference in etching effect can be found between Oberhoffer's and Fry's reagents, but that Fry's No. 2 is, in practice, the easiest and most convenient to use, because it does not require so well prepared a test-surface as Oberhoffer's reagent. Fry's No. 2 reagent is, likewise, as little suited to microscopic etching as the latter.

Canfield has proposed the following etching reagent—

Cupric chloride .	1·5 grams.
Nickel nitrate, cryst.	5 grams.
Ferric chloride .	6 grams.
Water . . .	12 c.c.

A few c.c. of nitric acid may be added if desired. The rate of etching can be controlled by altering the amount of water, and, if a slow etch is required, 150 c.c. of methyl or ethyl alcohol are added.

This is used for showing phosphorus segregations. The specimen is immersed in the solution; generally 90 seconds is an adequate period for etching, though occasionally several minutes are required. The metal which is deposited on the purer areas of the surface of the specimen shows colours varying from red-brown to pale brown. Segregations, particularly of phosphorus, appear quite white.

As an application of this etching reagent Canfield has worked out a method of "phosphorus-printing." Photographic paper is soaked for a few minutes in a 5 per cent solution of potassium ferricyanide in water. The paper is taken out and pressed for about 1 minute into contact with the surface which has been previously etched with the above reagent. Then, after the paper has been washed and fixed in the usual way, an impression remains of the areas which are contaminated mainly with phosphorus. Investigation has, however, shown that it is occasionally very difficult to obtain a clear impression, and the method, therefore, seems to call for very considerable experience. For "phosphorus printing" only a light preliminary etch is required.

Macro-etching for Showing Cold Working and Deformation Lines. (*Luder's lines*). B. Strauss observed in 1905 that on etching a piece of cold worked boiler-plate certain "lines" appeared. Fischer in 1913 found similar lines near the sheared edge of an iron plate which served as the anode in an electrolytic bath.

It was, however, through Fry's work, 1921, that more accurate details first became known as regards the possibility of etching up the so called "strain lines" in material which had been exposed to strains beyond the elastic limit.

For etching, Fry described two solutions, the first for microscopic use—

Cupric chloride	5 grams.	Fry's No. 3 etching reagent.
Hydrochloric acid . .	40 c.c.	
Water	30 c.c.	
Ethyl alcohol . . .	25 c.c.	

and the second for macroscopic work-

Cupric chloride .	90 grams.	Fry's No. 4 etching reagent.
Hydrochloric acid	120 c.c.	
Water . .	100 c.c.	

Both these etching solutions differ from the copper-containing reagents described previously, in that the concentration of HCl is so high that copper does not precipitate under any circumstances during the actual etching (see pages 15 and 52).

Before etching it is essential for the test-pieces to be heated up to 200–250° C. for about half an hour. After this heat-treatment the preparation of the specimens is carried out as usual. The ground surface—it need not be polished—is etched for 1–3 minutes in the solution, then the specimen is taken out and the surface rubbed with powdered cupric chloride by means of a piece of cloth for from 2 to 20 minutes. When the lines appear clearly the etching is stopped, the specimen wiped somewhat, then washed with alcohol, and dried as rapidly as possible. The washing must not be done with water because this causes copper to be deposited on the specimen. If the surface appears dirty it may be wiped over with a cloth saturated with the etching solution, and subsequently washed again in alcohol.

It has been found an advantage to rinse the specimen with fairly concentrated hydrochloric acid at the conclusion of etching. Washing with water can then be done without any copper being deposited.

In addition to bringing out strain lines, this etching reagent is also very suitable for ordinary grain-contrast etching, and is, furthermore, very often a good substitute for Oberhoffer's reagent when alloy steels are concerned.

Meyer and Eichholz modified the composition of the macro-etching solution by diminishing the HCl-content, and changing a part of the copper for ferric chloride—

Cupric chloride	20 grams.	Meyer & Eichholz' No. 1 etching reagent.
Ferric chloride	20 grams.	
Hydrochloric acid	30 c.c.	
Water	100 c.c.	

With this reagent the subsequent rubbing with cupric chloride was found to be unnecessary.

Later they changed over to a more dilute solution—

Cupric chloride	6 grams.	Meyer & Eichholz' No. 2 etching reagent.
Ferric chloride	6 grams.	
Hydrochloric acid	10 c.c.	
Ethyl alcohol	100 c.c.	

With this good results were obtained, even if the amount of hydrochloric acid was reduced to half. These weaker solutions require a somewhat longer time for etching than Fry's. They do not produce such strong contrasts but, on the other hand, are stated to bring out finer details.

As regards the temperature of tempering Meyer and Eichholz found that, with their more dilute solutions, the deformation lines could be etched up even after tempering at 50° C., while this was not possible with Fry's reagent. On tempering to over 300° C. the lines began to fade more and more. It was observed that the more rapidly deformation occurred, the higher was the temperature at which the specimen could be tempered without the lines disappearing.

Concerning these etching processes Jevons (1) reports as follows. Fry's method of etching is at present only suitable for soft iron and mild steel, while even with these materials uniform results are not always obtained. Thus Jevons did not succeed in obtaining any etching effect on an English steel of nearly the same analysis as the one used by Fry at Krupp's. After 20 hours etching only traces of deformation lines appeared. A carbon content of less than 0·3 per cent is essential for the etching to be possible. Steel with a pronounced fibre-structure gave a worse result than those without. The previous heat-treatment is believed to have a considerable influence on the result of etching. On the other hand, percentages of silicon, manganese, sulphur, or phosphorus within the usual limits, do not have any effect.

According to Jevons, the solutions of Meyer and Eichholz are not very suitable for etching up strain lines because they accentuate segregations too much, with the result that the clearness of the lines is diminished. Heating at from 200° to 400° C. does not seem to produce any noteworthy change in the result of the etching, but temperings at 100° and 600° C. respectively gave very poor results. A fairly copious supply of the solution must be available for etching because it is soon spent. During use the colour changes from green to brown: when it becomes sepia-toned the action proceeds so slowly that it is best to change the solution. Different steels require different quantities of the etching reagent, but as a rough approximation, 1 litre of

Fry's No. 4 solution may be allowed for every 2 sq. decimetres of the surface exposed in the bath. If the action is continued long many fine details are entirely lost, though a relief as high as 1 mm. can be obtained after one week's etching. With the above solution Jevons obtained good results after etching for 60–80 hours, provided the bath was kept well stirred. He did not find any advantage in the rubbing with cupric chloride.

Wazau preferred to etch the specimen first with cold strong hydrochloric acid for from 20 minutes to 3 hours, depending on the composition of the material, and pointed out, in connection herewith, that at very low temperatures—when the acid was partly frozen and the time of etching considerably prolonged—very fine details were brought out. After the hydrochloric acid etch the specimen was washed with water and Fry's reagent then applied with a brush, because, according to this investigator, the free access of air plays an important part. Wazau found, in agreement with Oberhoffer and Toussaint, that the lower the temperature of the etching reagent, the finer were the details which could be etched up. However, under such circumstances, the time of attack is prolonged, and the best compromise appears to be effected by etching at room temperature.

Wazau worked with slightly modified varieties of Fry's solutions and found that, with the following reagent for example, the specimen could be washed directly in water without risk of deposition of copper.

Cupric chloride	45 grams.
Hydrochloric acid	180 c.c.
Water . .	100 c.c.

With this modification somewhat greater contrasts in the etching effect were also obtained. If the etched surfaces showed contrasts too weak for photography Wazau allowed the specimens to lie in the air and rust slightly, subsequently cleaning them up on fine emery paper. As regards the preparation of the specimens it was found expedient to finish with a fine milling-cut, and then to etch the specimen for a few minutes in dilute acid. This method avoided the tedious filing and grinding.

After etching with Fry's reagents, 50 per cent HCl at about 50° C. can be used for the first washing, followed by water as usual.[1] By subsequent careful polishing the lines can often be caused to appear more clearly.

Macro-methods for Indicating Sulphur and Its Distribution. The methods which have been proposed for the macroscopic indication of sulphur in iron and steel are based on the fact that the sulphide inclusions occurring in the surface of the specimen evolve sulphuretted hydrogen under the action of acids. This sulphuretted hydrogen is caused to react with metallic salts which are held in contact with the surface by a suitable support, and which are of such a nature that a characteristic colour appears wherever any action occurs. The salts employed are those of mercury and silver, and the support for them consists of silk or gelatined paper, etc.

Heyn's Method of "Sulphur Printing." In 1906 Heyn published the following process which was worked out at the Materialprufungsamt in Berlin, in conjunction with the cupric ammonium chloride method of etching.[2]

A piece of silk is laid on the surface of the specimen, which needs no special preparation beyond filing flat, and is moistened, by means of a paint brush or some cotton wool, with the following solution—

Mercuric chloride	10 grams.
Hydrochloric acid	20 c.c.
Water	100 c.c.

From those places where the sulphide inclusions occur, sulphuretted hydrogen is evolved, and this, reacting with the mercuric chloride, precipitates black

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[1] Communication from a Swedish works.

[2] This account is based on a somewhat later, improved description by Heyn and Bauer.

TABLE IV

COMPARATIVE LIST OF THE ETCHING REAGENTS CONTAINING COPPER

	Heyn	Humfrey	Whiteley	Dickenson	Rosenhain and Haughton	Stead (1)	Stead (2)	Com-stock	Le Chatelier (1)	Le Chatelier (2)
H_2O c.c.	1,000	1,000	1,000	1,000	1,000	10?	200	?	180	100
$CH_3 . CH_2OH$ c.c.	—	—	—	—	—	900	1,000	950	1,000	1,000
$CuCl_2 . 2H_2O$ grm.	51	74	1·2	6	1	10	50	10	10	10
NH_4Cl grm.	32	46	—	—	—	—	—	—	—	—
$FeCl_3 . 6H_2O$ grm.	—	—	200	80	30	—	—	—	—	—
HCl, conc., c.c.	—	50	100	80	100	20	10	20	20	13–25
$SnCl_2 . 2H_2O$ grm.	—	—	—	—	0·5	—	—	—	—	—
$MgCl_2 . 6H_2O$ grm.	—	—	—	—	40	40	40	—	40	—
Picric acid, grm.	—	—	—	—	—	—	—	—	—	5
$Ni(NO_3)_2 . 6H_2O$ grm.	—	—	—	—	—	—	—	—	—	—

	Oberhoffer	Fry (1)	Fry (2)	Canfield	Fry (3)	Fry (4)	Meyer & Eichholz (1)	Meyer & Eichholz (2)	Wazau
H_2O c.c.	500	480	400	960	300	500	1,000	—	400
$CH_3 . CH_2OH$ c.c.	500	150	200	—	250	—	—	1,000	—
$CuCl_2 . 2H_2O$ grm.	1	8	4	12	50	450	200	60	180
NH_4Cl grm.	—	—	—	—	—	—	—	—	—
$FeCl_3 . 6H_2O$ grm.	30	—	—	48	—	—	200	60	—
HCl conc., c.c.	55	150	200	—	400	600	300	100	720
$SnCl_2 . 2H_2O$ grm.	0·5	—	—	—	—	—	—	—	—
$MgCl_2 . 6H_2O$ grm.	—	—	—	—	—	—	—	—	—
Picric acid, grm.	—	—	—	—	—	—	—	—	—
$Ni(NO_3)_2 . 6H_2O$ grm.	—	—	—	40	—	—	—	—	—

HgS, which remains fixed in the silk. A contact period of 4 to 5 minutes is usually sufficient. Owing to the vigorous evolution of hydrogen, bubbles are easily formed under the silk, but these must be carefully pressed down with the brush, which is kept filled with fresh solution. During the whole time of the test the silk must be kept in close contact with the specimen, and, from time to time, the weakened solution should be renewed by brushing over with fresh. At the end of the action the piece of silk is lifted off, and washed with water. If the latter is done carefully the HgS remains quite firmly fixed, and nothing is lost. The piece of silk now presents a true mirror-image of those places in the ground surface where concentrations of sulphur may be found. The method gives good results in all cases where local aggregates of numerous sulphide inclusions occur, but, if the sulphur is only present in small amounts, and is uniformly distributed throughout the section, there will only be a general grey-colouring of the silk, without any particular areas appearing darker. Phosphorus-rich areas, under the action of the acid, give off phosphoretted hydrogen which precipitates the lemon-yellow mercury phosphide from the mercuric chloride solution. Confusion with the black sulphide is, therefore, precluded. The evolution of phosphoretted hydrogen is, however, slow in comparison with that of sulphuretted hydrogen.

It may be mentioned in addition that, in the case of rather small sections, there is usually some difficulty in keeping the silk flat and in one position, particularly when painting it with fresh solution. It facilitates matters very much to stretch the silk in an "embroidery frame" of the kind which is commonly used on sewing machines. The piece of silk should remain in the frame until it is quite dry, as otherwise it may shrink.

Royen and Ammermann's Method. Royen and Ammermann have improved Heyn's method by using gelatined paper instead of silk. The paper is soaked for 2 minutes in Heyn's solution, dried superficially, then laid on the surface of the specimen, and lightly pressed close by stroking, not more than twice, with the edge of a piece of cardboard. The contract time is 3 to 5 minutes. The paper is then pulled off, and carefully wiped under water with cotton wool to remove a dark film which usually forms. Finally, it is washed for 1 hour in running water, and dried in the usual way, using a glazing-plate if desired.

Bromide-paper, from which all silver has been dissolved out with fixing-salt, can be used, otherwise Messrs. Bayer, of Leverkusen, Germany, prepare a special paper which is suitable for this purpose. If the paper is dried after soaking in the acid mercuric chloride solution, it may be stored in this condition and then, before use, merely dipped in dilute hydrochloric acid (20 c.c. HCl, s.g. 1·12, in 100 c.c. water).

Royen and Ammermann suggest that the above process should be called "sulphur printing," and that the Baumann method (see below) should be described as "Baumann printing." It is further recommended that a Baumann print be made first, and then, after regrinding the surface, a "sulphur print," subsequently comparing the results. The effect with the "sulphur print" is identical in every respect with Heyn's original method but gives a sharper and clearer print.

The colour of the Baumann print is, in general, brown. This changes to a much darker, almost black, colour in small areas which are often in the form of points or lines. The mercuric-chloride prints are valuable for determining the relative importance of these dark-coloured areas because, in this latter type of print, any phosphorus is indicated by pale lemon-yellow areas.

The Baumann Print. This method was published by Baumann in 1906, shortly after Heyn's first communication, and was briefly described as follows.

A simple method of determining the points of sulphur concentration in the surface of a specimen may be based on the effect on a silver salt of the sulphuretted hydrogen evolved with acids. An ordinary photographic paper is soaked with dilute sulphuric acid and lightly wiped. Then, with the film side inwards, it is pressed carefully against the test surface which has been previously ground flat and cleaned from grease. When, after a short time, the paper is pulled off again, the sulphur-containing areas appear dark coloured in the contact print so obtained, while the areas free from sulphur remain white, or only slightly shaded. The paper is then fixed and washed in the ordinary way. Several prints may be obtained from the same surface, and it may be mentioned that a dark-room is not necessary. In a footnote Baumann refers to Heyn's process, but considers that the method using silver bromide paper is simpler and gives sharper pictures.

Quite a vigorous discussion then took place as regards the merits of the Baumann print compared with Heyn's sulphur print (Baumann, Heyn, Bauer, Oberhoffer (4); Oberhoffer and Knipping, and others). The following is an abstract of what accrued during the discussion, amplified with some of the writer's own experience.

Technique of the Baumann Print. The photographic paper should be thin, so that good contact is easily obtained. Glossy paper gives sharper prints, but, on the other hand, is harder to manipulate, particularly when printing from small specimens, because it slips extremely easily on the surface. Much annoyance due to the latter cause can be avoided if a matt paper with a smooth surface is chosen.[1] The paper is soaked for 2 minutes in sulphuric acid solution. With a shorter time of immersion it does not absorb enough solution, while with a longer the gelatine swells too much, and becomes slippery, so that the paper slides about on the surface of the specimen. When the paper is taken out of the sulphuric acid, it is laid on a glass plate, or some flat support, with the film side up, and the excess of solution allowed to drain off for a few moments by holding the paper on its support at an angle. Then, taking a chamois leather, which has been wrung out with water, or alternatively some cotton wool or a sponge similarly treated, the gelatine surface is lightly and evenly wiped, so that no solution remains except that which has been absorbed in the gelatine. The paper is now loosened from the support and laid in contact with the surface which has been prepared. For small surfaces of a few square centimetres area it is simplest to hold the specimen in the hand, and press the paper tight with the thumb. For medium-sized sections the specimen is laid on a firm support with the prepared surface upwards, the paper being laid on this, and pressed with the flat of the hand, or with a ball of wool or cloth. As in Royen and Ammermann's method, stroking with the edge of a cardboard sheet is often an excellent expedient.

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[1] Kodak contrast bromide paper is an excellent quality for Baumann prints.

For large surfaces, such as complete ingot sections, etc., the paper may be conveniently rolled up on a roller or the like. The free end of the paper is then suitably fixed just overlapping one of the short edges of the section, and the remainder of the sheet then rolled out over the surface, at the same time pressing it tightly against the latter with a *soft* rubber roller, so that all air-bubbles are removed. Whatever method is used it is important for the paper to be kept in intimate contact with the entire surface of the specimen for the whole period of printing. Owing to the evolution of hydrogen, gas-bubbles are easily formed between the paper and specimen, and they tend to lift the paper off. These bubbles must be immediately stroked out to the nearest edge. In critical cases the paper can be pierced with a pin. If a particularly long contact-time is necessary, it may be useful to moisten the back of the paper with the sulphuric acid employed previously, or even with water, so that the paper will not dry up, and possibly stick fast. For surfaces of ordinary size very good contact can be obtained by pressing the paper and specimen together in a letter press for example, or a Brinell machine, or in some similar way. This method provides exceedingly sharp and clear prints, but requires practice to obtain successful results. Under too great a stress the gelatine is pressed asunder, and the paper sticks, while, if the pressure is too light, gas-bubbles easily form between the specimen and paper and prevent contact locally. After a suitable time the paper is carefully pulled off. If it should have stuck fast, the specimen and paper are immersed in water, and, as a rule, the paper can then be loosened. Care should be taken that, when the paper is laid on the specimen, the latter is not at a higher temperature than the room, otherwise the former will stick infallibly and irretrievably. After being taken off, the paper is washed in water for 5 to 10 minutes, and then fixed in a neutral bath; an acid fixing bath attacks, to a certain extent, the silver sulphide which has been formed. The final washing and drying is carried out as usual for photographic paper.

Preparation of the Specimen. Baumann printing can be carried out quite well on a relatively rough surface, e.g. directly on a surface which has been filed flat, or planed. If it is desired to bring out as much detail as possible, the surface must be finely ground. The grinding should be done with a certain amount of care, so that the usually brittle sulphide particles do not crumble and fall out, as then, of course, a poor print will be obtained. On the other hand, polishing is unsuitable because the paper slides about very easily on a surface so prepared. The latter must be made quite clean, and free from grease, before the paper is laid on it.

A Baumann print of a machine-prepared surface often shows the marks on the steel if considerable cold-working has occurred in the underlying material.

Concentration of the Sulphuric Acid Solution. In the literature sulphuric acid solutions of concentrations varying from 1 to 10 per cent are recommended. Too low a concentration of acid involves a certain risk that metallic silver will be reduced out owing to the action of the iron. A photographic paper, moistened with water, is blackened in this manner after a sufficiently long time in contact with pure iron. Papers containing silver chloride are blackened more easily, owing to the reduction of the silver, than those containing only silver bromide. On the other hand, a 10 per cent sulphuric acid solution acts somewhat destructively on the gelatine film, and makes it loose and slippery. A 5 per cent solution can be recommended as being generally the best, because it does not give rise to any serious complications of the kind mentioned. When the percentages of sulphur are particularly high the strength of the acid should be somewhat diminished.

Time of Contact. Contact times of from 1 to 10 minutes are used. It is often recommended to make the time inversely proportional to the sulphur content of the specimen. Thus the A.S.T.M. recommends a contact time of 6 minutes for a sulphur content of 0·028 per cent, and 45 seconds for 0·18 per cent. According to the writer's experience, however, it seems to be best, in practice, always to employ a constant time of contact of 3 or 4 minutes, and only to depart from this by way of exception. In this way more easily comparable results are obtained.

When taking prints from entire ingot-sections a longer time is required, as a rule, because, in such cases the contact between section and paper is inferior to that obtained with smaller surfaces. Ten minutes is generally a suitable period.

Interpretation of the Result. The sulphuretted hydrogen evolved from each sulphide inclusion occurring in the surface of the specimen reacts locally with the silver bromide paper. Nevertheless, diffusion always occurs in the gelatine film, and the blackened areas on the paper will, for small particles, be possibly 5 to 10 times the actual diameter of the sulphide inclusions. If a Baumann print is examined under moderate magnification the separate small blackened areas can be distinctly made out, although to the unaided eye the effect often appears to be a uniform darkening of the paper over a large or small area. Primrose considers that the particles which make impressions in the Baumann print seldom contain more than 1–2 per cent sulphur. With cracked, or porous, materials, misleading results are frequently obtained because of the locally increased areas open to the attack of the acid.

A point worth considering is that different photographic papers have different compositions and percentages of silver, and this influences the intensity of the blackening.

Schottky observed that the Baumann prints from longitudinal and transverse sections of the same material had a characteristic difference. In the transverse section a considerably more intense blackening of the paper occurred than in the case of the longitudinal. Schottky utilized this effect for determining the direction of rolling in plates.

Concerning the question whether the Baumann print indicates sulphur only, or includes phosphorus as well, it can be safely said that under normal conditions as regards material, acidity, paper, and contact time, it shows sulphur to a preponderating extent. Only in very exceptional cases does the phosphorus affect the result.

Without making further tests it is not possible to determine from a Baumann print the real or relative magnitudes of the percentages of sulphur indicated, because the intensity of colour does not depend on the latter alone. The same percentage of sulphur yields lighter prints with low percentages of carbon than with high. According to Durand the same thing holds for manganese. The higher the manganese content the darker the Baumann print. For nickel- and chromium-steel the method is perfectly useless, because it does not give any evidence at all of the sulphur content. A heavily cold-worked surface gives darker prints than one not cold-worked.

A specimen containing a high percentage of sulphur always gives a dark, vigorous print. The reverse, however, is not always true, because a relatively dark Baumann print need not always signify high sulphur content, even though this may be so in the great majority of cases.

Baumann Printing on Photographic Plates or Films. Durand, 1924, carried out Baumann printing with photographic plates and films instead of with silver bromide paper. This method has the advantage that the transparent print can be laid on, and compared with, the macroscopically etched surface.

Tischy made a number of tests of Durand's suggestion, and found that the distinctness with which the details appear depends directly on the degree of smoothness of the surface of the specimen. With rough preparation of the latter only diffuse coloration is obtained in those places where sulphide particles are collected. With better preparation of the specimen the details appear so distinctly that a picture, enlarged about fifty times, can be prepared, and compared with a photomicrograph of the etched surface at the same magnification. If a certain most favourable time of contact is exceeded the details of the print lose their sharpness owing to diffusion. For a material containing

0·1 per cent C and 0·03 per cent S a good result was obtained with a film after a contact of 1 minute using 5 per cent sulphuric acid.

Rogers' Method of Obtaining Baumann Prints of Fractures. Rogers, who attempted to prepare Baumann prints direct from fractured surfaces, published the following method in 1912.

450 grams of vaseline are melted together with about 50 grams of wax, and of this mixture the smallest possible quantity is worked up with whiting, so that a stiff, coherent, plastic mass is obtained. This mass is rolled out into a sheet of about 2 mm. thickness. Plasticine is too soft for this purpose, and has an unsuitable colour; it can be used, however, if it is impregnated with whiting.

For the silver bromide emulsion digest the following at about 43° C.: water 150 c.c., gelatine 15 grams, 10 per cent HCl 3 c.c., and potassium bromide 8·4 grams. Then add the following solution, similarly heated to 43° C.: water 50 c.c., silver nitrate 10 grams. The whole is kept at 43° C. for about quarter of an hour. Next, cool quickly and allow the mass to set. The latter is then broken up, tied up in an open textured cloth bag, and washed in running water for at least two hours. The bag is then taken out and the water allowed to drain off. Finally, the mass is emptied out and melted up again with the addition of a further 15 grams of gelatine. The emulsion so obtained is now ready to be joined on to its support.

Warm up a sheet of the plastic material to not more than 40° C., and dust the surface with whiting. Rub the surface carefully till all traces of grease have disappeared from it. Dust away the excess of the whiting, and then pour on the emulsion. Its spreading can be assisted with a glass rod, and by tilting the sheet. At the end of this process lay the sheet horizontal in a dust-free place to dry.

In order to obtain a print allow a prepared sheet to soak for 20–30 seconds in a solution of 100 c.c. water, 1 c.c. sulphuric acid, and 5 grams alum. See that the surface is well moistened, and then press the sheet into contact with the fracture with the fingers. Loosen the print after about 12 seconds, or longer if the steel has a very low sulphur content. For larger surfaces it is best to make the prints in sections. Fixing may be done in the usual way, but, as there is a certain risk involved of the emulsion coming away from the sheet, this stage is generally omitted. It is sufficient to wash the print carefully for a few minutes, and then allow it to dry. The solution and the steel should not be hotter than 20° C., as otherwise the gelatine may easily melt. A little practice is necessary to learn the printing technique.

The prints taken direct from fractures are considerably darker than those from sectional surfaces.

In 1919 Portevin tried to carry out Rogers' method, but was not successful because the emulsion came away from the support. After various trials with other supporting media he found that hectograph jelly formed a suitable plastic base from which the emulsion did not come loose. (Roller composition, obtainable from printers, is probably still better).

Plastic mass	1 part gelatine 2 parts water 4 parts glycerine

The gelatine[1] is allowed to swell in the water, then melted on a water-bath and the glycerine added. To make the mass opaque, so that the contrasts in the print appear stronger, 50 per cent of barium sulphate can then be added, but this is not really essential.

Silver Bromide Emulsion—

Solution A	Water	30 c.c.
	Gelatine . . .	3 grams.
	Hydrochloric acid . .	1 drop
	Potassium bromide .	1·6 grams.

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[1] It is possible to use glue instead of the gelatine.

The gelatine is dissolved in the water on a water-bath, then the other additions are made.

Solution B	Water .	15 c.c.
	Silver nitrate	2 grams.

Solution B is poured into A, the mixture stirred, and then cooled in a large clock-glass or a photographic dish. When it sets it is cut up with a horn-bladed knife or the like into about 1 cm. strips, which are then subjected to a thorough washing. This can be conveniently done by putting the emulsion into a porcelain or earthenware teapot, which is closed with a layer of thin cloth. The spout is then connected to the water supply with a piece of rubber-tubing, and a slow stream is turned on overnight. If the supply contains high percentages of iron or chalk the washing must be done with distilled water. At the end of this stage the emulsion is taken out, allowed to drain, then remelted on a water bath, with the addition of a further 3 grams of gelatine, and finally poured over the surface of the hectograph jelly. When the emulsion has dried a piece of the jelly, suitable for the surface of the specimen, is cut off and soaked for 2 minutes in 3 per cent sulphuric acid. Finally it is pressed into contact with the fracture for 1 minute.

Macro-methods for Studying Cracks. While it is true that cracks can be shown up by such etching reagents as strong acids, etc., yet, as a rule, with these methods, they appear on a considerably exaggerated scale. Moreover there is always a risk that, particularly in the case of really deep etching, cracks may form during the etching itself if the material is not entirely free from strain.

It is reported[1] that treatment in saturated copper ammonium chloride solution (*cf.* page 52) has been found very useful for discovering defects due to cracks, e.g. "flakes." This method of etching does not widen the cracks to any great extent, as is shown by the fact that, afterwards, they are often so extremely fine that it is necessary to use a lens to see them plainly. The specimen should have a fairly fine structure, and is, therefore, annealed or quenched and fully tempered before treatment.

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[1] From a Swedish works.

Keshian recommends a 10–15 per cent solution of nitric acid in water or alcohol for etching up fine cracks.

Homerberg employs a more superficial etching with a reagent to which some strong colouring matter is added. After etching the surface is rapidly dried and then watched for the reappearance of liquid as the specimen is warmed up.

Hoke and others at the Bureau of Standards (1919) worked out the following magnetic method for investigations on fine cracks.

The specimen, which must be as well polished as for microscopic work, is magnetized, and then immersed in a dish of kerosene containing fine iron-dust in suspension. The "cast-iron mud" from lapping discs is very suitable for the latter. The iron dust collects along the cracks because they cause discontinuities in the magnetic field. The specimen is subsequently washed in clean kerosene to free it from the excess of dust.

CHAPTER IV

MICRO-ETCHING OF IRON AND STEEL

Introduction. In the micro-etching of iron and steel two different types of etching have to be considered, namely, etching to develop the general structure, and selective etching to show up any carbides present. The former type, which, in what follows, is designated "structure etching," is carried out, as a rule, with acid etching reagents, e.g. nitric or picric acid, and by this means the grain-structure of the material is made visible. The latter type, which is subsequently called "carbide etching," is done with alkaline solutions, which are usually mixed with an oxidizing agent, such as hydrogen peroxide, potassium ferricyanide, or potassium permanganate; electrolytic etching is also used occasionally. The carbides which occur are coloured in a characteristic manner, but the other grain structure of the material does not generally appear. According to Goerens (2) the size of the carbide particles has a definite influence on the etching result: thus, the cementite lamellae in pearlite are not coloured if their thickness falls below a certain limit.

ETCHING OF IRON AND LOW CARBON STEEL

The grinding and polishing of iron and low carbon steel requires great care because the surface very easily becomes cold-worked to a considerable depth, making it difficult or impossible for the true structure to appear on etching. Fig. 5 shows a soft iron which was prepared by hand-grinding on emery paper. After polishing the surface appeared to be flawless, but, on etching, the cold-worked structure was disclosed and this more or less obliterated the grain-boundaries beneath. Fig. 6

shows the same material ground on a lead disc with emery and oil, but polished in the same way as the previous example. The structure here appears much clearer, but even yet not quite free from flaws.

According to Palmer, if the specimens are etched immediately after polishing the result is often inferior to that obtained if they are allowed to stand 24–48 hours beforehand.

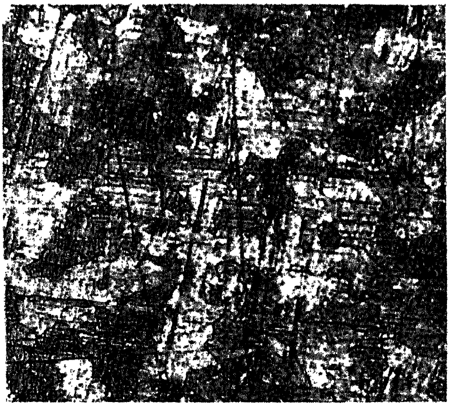

FIG. 5. SOFT IRON ETCHED FOR 30 SECONDS IN 4 PER CENT HNO_3 IN AMYL ALCOHOL AT 35° C.
Grain structure partially obliterated by "latent" grinding scratches; × 50

The effects of the cold-worked layer formed during grinding and polishing can be partly eliminated by alternate etching and polishing. In using this method care should be taken that the first etching is relatively deep, and that subsequent attacks are successively diminished. The etching can be done equally well with picric or nitric acid dissolved in ethyl alcohol. With the latter acid, however, pits are easily formed round any non-metallic inclusions which may be present. According to a method worked out by the writer[1] this effect can be avoided if the nitric acid is used in amyl alcohol. The etching is carried out in a

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[1] At the Government Testing Institute, Stockholm.

4 per cent solution kept at a temperature of about 70 C., and the specimen is also heated to this temperature beforehand. The heating is best done by placing the etching-dish and the specimen together on an electric hot-plate provided with a variable rheostat. The first etching, for 40–60 seconds, is carried out, with advantage previous to polishing, then the specimen is reground with the finest available medium, and polished. (A comparatively strong etching previous to polishing, in order to eliminate grinding scratches is also recommended by several other writers: Pulsifer, Homerberg, and others.) The etching is carried out again for 30 seconds, followed by repolishing. The process is repeated for the necessary number of times, and this, after some experience, can be judged from the appearance of the etched surface. With a carefully ground specimen about three etchings should be sufficient to obtain a perfect surface. The last etching is made somewhat shorter than the previous ones, e.g. it may be continued for about 20 seconds, and then the specimen is finally polished.

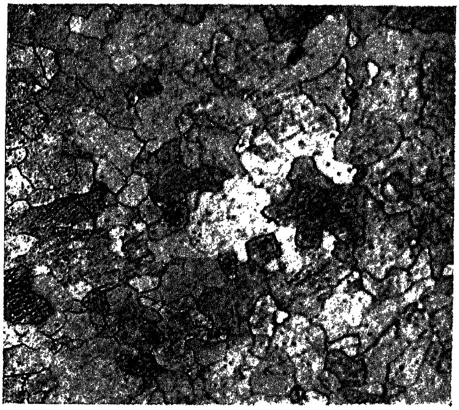

FIG. 6. SOFT IRON ETCHED AS IN FIG. 5

Improved appearance of the structure as a result of more careful preparation of the specimen

Picric Acid. 4 per cent, up to saturated, solutions of picric acid in alcohol are very suitable for clear grain-boundary etching of ferrite. The time of etching is, however, relatively long, 2–10 minutes, and it is necessary for the surface of the specimen to be brushed continuously in order to obtain an even attack. Campbell recommends etching for 1 minute for showing pearlite in wrought iron, and for 5 minutes for bringing up the grain boundaries.

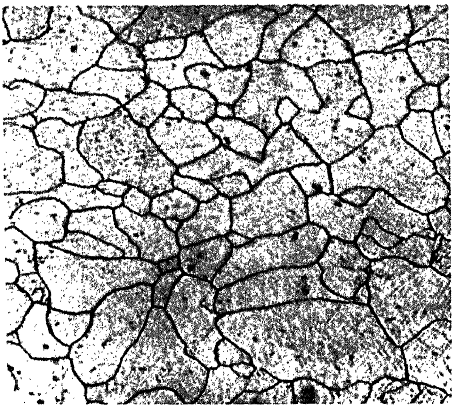

FIG. 7. GRAIN-BOUNDARY ETCHING OF FERRITE

Etched with saturated solution of picric acid in ethyl alcohol. Time, 3 min., at room temperature; × 100

The action of picric acid on ferrite appears to be that the iron is dissolved evenly and regularly from the surfaces of the various grains, but with different speeds for the individual grains, depending on the crystal orientation. As a result the surfaces of the grains will lie in various planes, which means that, under observation at high magnification, the grain-boundaries will not all appear alike, and that, furthermore, they will be fairly thick. For magnifications up to 200×, however, the picric acid etch is very suitable and, within the grains, many fine details appear, which would be lost by using other reagents.

The rate of attack of the etching reagent depends to some extent on the grain-size of the material and its previous heat treatment. Thus the smaller the grain-size, the longer is the etching time. Fig. 7 shows an example of the etching of ferrite with picric acid.

Nitric Acid. A 2 per cent solution in ethyl alcohol may be used with advantage for etching soft iron. The time of etching, which varies from 10 seconds to 1 minute, depends on the grain-size and purity of the material. Prolonged etching strongly attacks the ferrite grains, and gives them a rough appearance; this is particularly the case with cold-worked material (even superficially cold-worked by grinding).

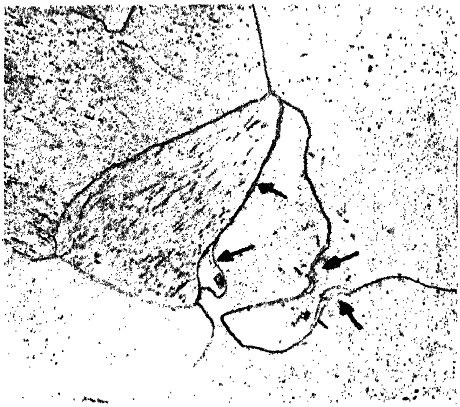

FIG. 8. SOFT IRON CONTAINING 0·02 PER CENT C
Cementite at grain-boundaries. Etched for 10 sec., in 1% HNO_3 in ethyl alcohol; × 500

For examining soft iron for the presence of free cementite a very short etching in a 1 or 2 per cent solution is recommended.[1] In this case a satisfactory etching time is usually 5–10 secs.; as a result, the grain-boundaries are only brought out very faintly, but the lines of demarcation between cementite and ferrite can be clearly made out at high magnifications. An example is given in Fig. 8.

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[1] Communication from A. Lundgren, metallurgical engineer, Government Testing Institute, Stockholm.

A 4 per cent solution of nitric acid in (iso-) amyl alcohol may be said to be, in general, the best etching reagent for soft iron. At room temperatures good grain-boundary etching is obtained after about 3 secs. to 1 minute. The roughening of the surfaces o the ferrite grains occurs much more seldom when using amyl- instead of ethyl-alcohol solutions.

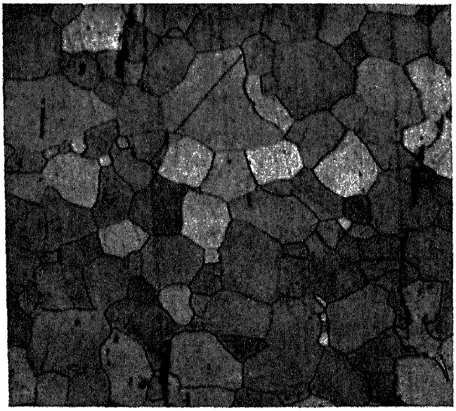

FIG. 9. FERRITE IN "LANCASHIRE" IRON, SHOWING PRONOUNCED GRAIN-BOUNDARY ETCHING BUT WEAK GRAIN-CONTRAST

Etched for 30 sec., in 4% HNO_3 in amyl alcohol at 25–30° C.; × 50

According to some results recently obtained[1] the etching effect developed by nitric acid in amyl alcohol is very closely connected with the temperature. At low temperatures this solution appears to have only a purely solvent action, which produces grain-boundary etching, while at higher temperatures it has a strongly oxidizing action, which results in grain-contrast etching, because of the reaction products adhering to the surface. The transition between the two types of action seems to occur at, or slightly above, ordinary room temperature. Thus, at 0° C., a brilliant grain-boundary etching is obtained. In this case the etching-dish is placed on snow or ice, and the specimen is also cooled before treatment. If moisture should condense on the cold polished surface, a few drops of ethyl alcohol are placed on it before the specimen is transferred to the amyl-alcohol solution, because the latter only dissolves water slightly. The time of etching varies between 2 and 10 minutes. An etching suitable for about 500 diams. magnification usually takes 3 minutes. After etching the surface should appear quite bright to the naked eye. A "matt" appearance indicates that the surface is cold-worked. If, finally, a weak grain-contrast etching is desired, suitable for photography, the specimen treated at 0° C. is subsequently etched for about 30 seconds in the solution heated to 30–35° C. (Figs. 9 and 10.)

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[1] At the Government Testing Institute, Stockholm, in connection with investigations for the Technical Committee of the Association of Swedish Ironmasters.

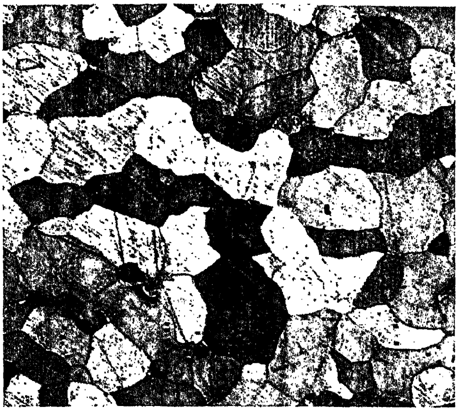

FIG. 10. FERRITE, SHOWING STRONG GRAIN-CONTRASTS WITH MODERATE GRAIN-BOUNDARY ETCHING

Etched for 30 sec. in 4% HNO_3 in amyl alcohol at 35–40° C.; × 50

On further increasing the temperature of the reagent the grain-contrast etching becomes more and more accentuated and, at 50° C., very strongly marked contrasts are obtained which, although very suitable for grain counting, and so on, are much too great for photography.

The etching at 70° C., mentioned in connection with the preparation of the specimen, causes the formation of a uniformly thick, fairly loosely adherent, surface film of oxidation products. Here the various grains can hardly be distinguished because, at this temperature, the action of the etching reagent appears to be relatively independent of crystal orientation.

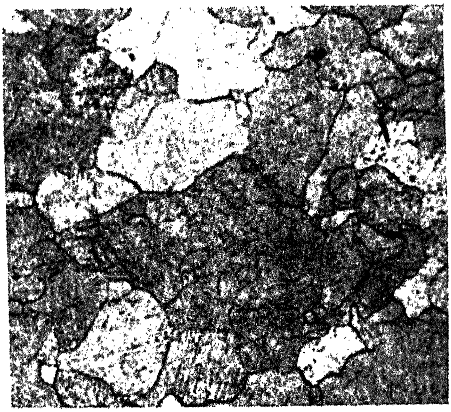

FIG. 11. FERRITE ETCHED AT ORDINARY ROOM TEMPERATURE
First for 3 min., with 5% picric acid in ethyl alcohol, and then for 10 sec., in 0·2% ammonium persulphate; × 100. (Bureau of Standards)

Tincture of Iodine, and Copper-ammonium Chloride. Tincture of iodine and copper-ammonium chloride are sometimes used for micro-etching, though they are definitely macro-reagents. Only a very short etching time is employed for micro-purposes. Nevertheless these etchants have no advantages over the others described here.

Ammonium Persulphate. Like the two foregoing reagents, ammonium persulphate is, strictly, a macro-etching medium (*cf.* page 47). In micro-work, however, according to Rawdon and Berglund, a very dilute solution is often useful for lightly colouring the various ferrite grains after etching with picric acid. For this purpose a 0·1–0·5 per cent solution in water is employed, and an etching time of 5–15 seconds. The solution should be cold or, at the most, not exceeding 30–35° C.; at higher temperatures it very easily causes staining, particularly if the material contains slag. Fig. 11 shows an example of this type of etching.

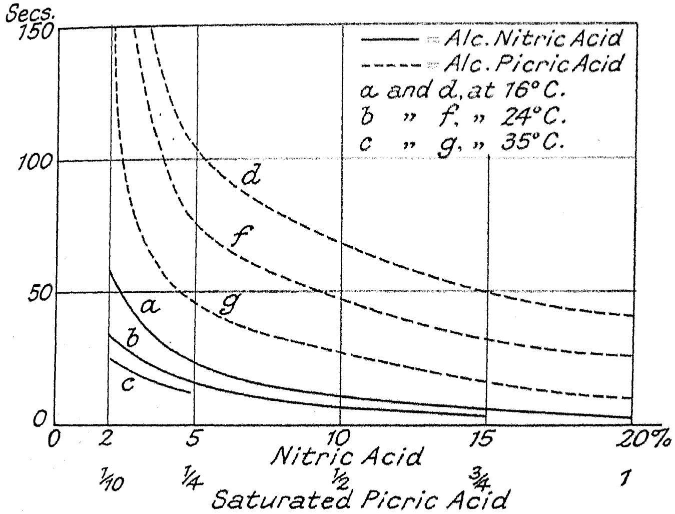

FIG. 12. THE EFFECT OF THE TEMPERATURE AND CONCENTRATION OF THE REAGENT ON THE ETCHING TIME OF PEARLITE IN 0·30 PER CENT CARBON STEEL
(Loskiewicz)

THE ETCHING OF NORMAL CARBON STEELS

Solutions of picric or nitric acid in ethyl alcohol are generally employed for the structure etching of normal[1] carbon steels.

Loskiewicz has carried out an investigation on the relation between the time of etching and the temperature and concentration of the reagent in the case of both the above etchants. Figs. 3 (page 21) and 12 represent the results obtained. As regards the influence of the carbon content of the material on the time of etching of the pearlite, Loskiewicz found that, at a temperature of 20° C., the etching time fell to about half as the carbon rose from 0·30 per cent to 0·90 per cent. As the latter increased still further up to 1·20 per cent the etching time then remained constant (Fig. 13).

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[1] The term "normal" is used here to mean normalized, annealed, etc. A later section is devoted to hardened and tempered steels.

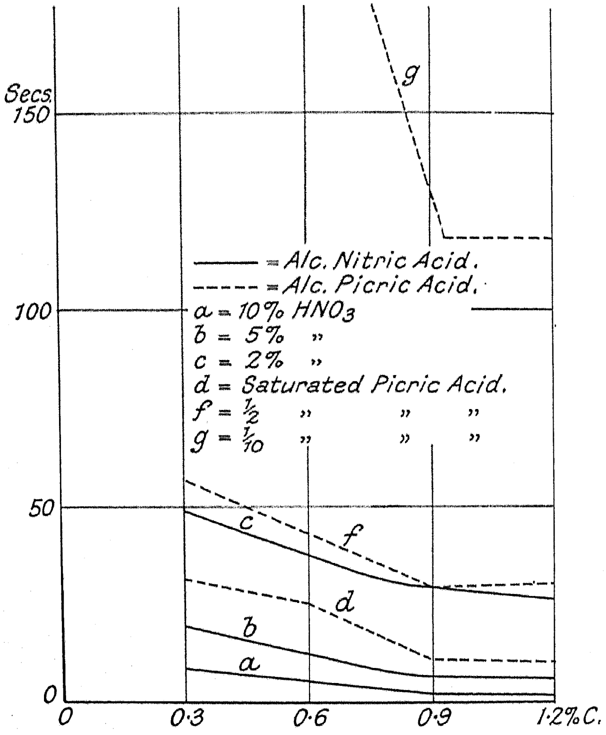

FIG. 13. THE EFFECT OF THE CARBON CONTENT OF THE STEEL ON THE ETCHING TIME OF PEARLITE AT 20° C. (Loskiewicz)

Picric Acid. Picric acid is used in 4 per cent, up to saturated, solutions in ethyl alcohol. It is reported[1] that the etching up of the grain boundaries is made easier by the addition of 1 per cent HNO_3. Picric acid seems to give a somewhat clearer etching than nitric acid when the material concerned has a very fine or indistinct structure.

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[1] Communication from a Swedish works.

According to the A.S.T.M.[1] a solution of picric acid in absolute ethyl alcohol is the best etching reagent for developing the structure of pearlite. Goerens (1), however, recommends amyl alcohol as the solvent.

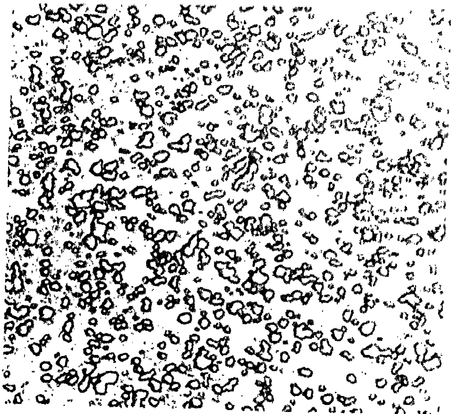

FIG. 14. FULLY ANNEALED STEEL CONTAINING 1·10 PER CENT C
Etched for 10 sec., at 18° C., in 4% HNO_3 in amyl alcohol, × 500

Nitric Acid. A 1 per cent solution in ethyl alcohol is probably the most usual, but 2, 3, 4, and 5 per cent solutions are also in general use. The weaker concentrations are best for etching the structure of the pearlite, while the stronger are more suitable for grain boundary etching. For this latter purpose up to 10 per cent solutions are sometimes used. To etch up the pearlite and the grain boundaries simultaneously, 3–4 per cent solutions are chiefly employed.

A mixture of equal parts of 1 per cent HNO_3 and saturated picric acid in ethyl alcohol is sometimes advantageous for grain-boundary etching.

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[1] *Proceedings*, 1923, I, 924.

Boylston[1] recommends a 5 per cent solution in absolute ethyl or methyl alcohol for the general etching of normal carbon steels. Nitric acid is more suitable than picric acid for etching ferrite occurring at grain-boundaries.

A solution of nitric acid in amyl alcohol, usually 4 per cent, generally etches more uniformly and somewhat more slowly than the corresponding ethyl solution, and it is preferred by the writer for all etching of carbon steels. Fig. 14 shows an example of spheroidized cementite etched with this reagent. For photomicrography, etching for 1 minute in the reagent cooled to 0° C. has been found to be an advantage for pearlitic steels because, in this method, the pearlite areas only acquire various shades of brown. At higher temperatures, and on etching in ethyl alcohol solutions, these areas often show variegated colours which give a faulty impression when photographed. Fig. 15 shows a steel

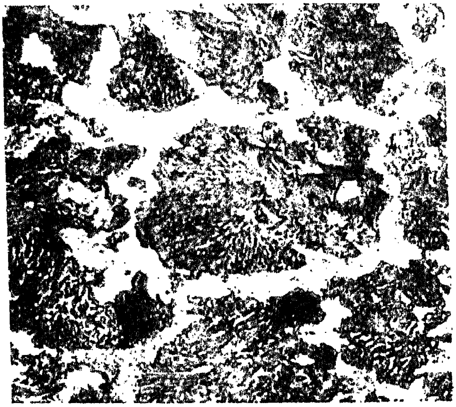

FIG. 15. ROLLED STEEL CONTAINING 0·55 PER CENT C.
Etched for 60 sec., at 0° C., in 4% HNO_3 in amyl alcohol × 500

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[1] *A.S.S.T. Handbook*, 1929.

containing 0·55 per cent. C. etched for 1 minute at 0° C. (The plate has been developed by, so called, divided development.)[1]

Etching in a solution heated to 50–60° C. is a very convenient way of showing free cementite in hyper-eutectoid steels, and also for observing small amounts of the former in soft iron. In this method of etching the ground-mass is darkened, but the cementite remains clear. Fig. 16 shows an example of this etching applied to a carburized material. The cementite network can be clearly observed.

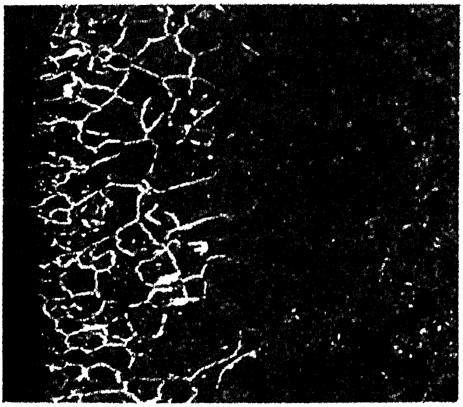

FIG. 16. CARBURIZED "ARMCO" IRON
Etched for 30 sec., at 55° C., in 4% HNO_3 in amyl alcohol,

Metanitrobenzol Sulphonic Acid. According to a communication received from de Reyna and Daernis, a 5 per cent solution in alcohol is used in the Altos Hornos iron-works, Viscaya, Spain, for unhardened steels. (For its use with hardened steel see page 99.) The pearlite appears blue, and with irridescent colours.

Iodine Solution. A solution of 5 grams iodine in 100 c.c. ethyl alcohol was introduced by Osmond[2] for etching iron and steel. Stead[1] recommends a solution of 1·25 grams potassium iodide and 1·25 grams iodine in 1·25 c.c. water, the mixture being made up to 100 c.c. with alcohol.

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[1] The plate is developed for one minute in a developer without alkali, and then transferred to another dish containing a solution of alkali in water.

[2] *A.S.S.T. Handbook*, 1929.

These etching reagents are seldom employed at the present time.

Potassium Bitartrate. Le Chatelier[1] has proposed a solution of potassium bitartrate for identifying ferrite, which is blackened by the reagent.

Carbide Etching. According to Groesbeck (3) iron carbide is darkened after 5–10 minutes etching in a boiling 5 per cent solution of potassium or sodium hydroxide. Stronger solutions blacken the cementite more quickly but, at the same time, discolour the ground mass. Long continued etching with a very strong solution, 50 per cent for example, results in the carbide being wholly or partly dissolved away, leaving holes behind. A cold solution does not colour the carbide in up to 30 minutes etching. Oxygen, whether led in as a gas, or "nascent" (e.g. from added hydrogen peroxide), seems to have no effect on the etching result.

Alkaline Sodium Picrate. This etching reagent was first proposed by Ischewsky. It is prepared by mixing equal parts of a cold saturated aqueous solution of picric acid, and a 50 per cent solution of caustic soda.

Another common method of preparation consists of adding 2 grams picric acid to a solution of 25 grams NaOH in 75 c.c. water, and warming the mixture until the picric acid is dissolved, then diluting with water to 100 c.c.

The solution is used nearly boiling, and the specimen is etched in it for 5–15 minutes. The liquid should be maintained at its original volume.

According to Loskiewicz a boiling solution of 5 grams picric acid and 20 grams NaOH in 100 c.c. water gives incipient colouring to cementite in 2 minutes, and a dark-brown shade in not more than 20 minutes.

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[1] *A.S.S.T. Handbook*, 1929.

According to Groesbeck (3) it seems that the action of the sodium picrate on cementite can be ascribed mainly to the presence of free alkali, because a neutral picrate solution (*cf.* page 118) does not blacken the carbide after 60 minutes etching in the nearly boiling liquid.

Alkaline Solution of Sodium Benzoate. Thompson and Whitehead state that iron carbide is blackened by a boiling alkaline solution of sodium benzoate, prepared by dissolving 6·3 grams benzoic anhydride and 20 grams NaOH in 100 c.c. water.

Groesbeck (3) finds that this solution behaves similarly to alkaline picrate solution, although more slowly. 30–45 minutes etching blackens coarsely laminated pearlite, but spheroidized cementite is only faintly coloured. Groesbeck attributes the slower action of the reagent to its lower alkalinity.

Alkaline Solution of Sodium Plumbate. Loskiewicz reports that a solution of 5 grams PbO and 20 grams NaOH in 100 c.c. water, acts in a similar way to the foregoing reagent.

For etching cementite, Thompson and Whitehead used a solution of equal parts of 10 per cent lead nitrate solution and 50 per cent caustic soda. This is described by them as Le Chatelier's carbide etching reagent. According to Groesbeck (3) the iron carbide is coloured after 5–10 minutes in the hot solution.

Alkaline Potassium Ferricyanide. Murakami's reagent.

Potassium ferricyanide	10 grams.
Sodium hydroxide .	10 grams.
Water . . .	100 c.c.

Cementite is coloured after 5–10 minutes in the boiling solution. However, other carbides occurring in steels are darkened or, more correctly, dissolved out, considerably more rapidly than the iron carbide (*cf.* page 108).

Daeves changed the amount of the ferricyanide as follows—

Potassium ferricyanide	20 grams.
Sodium hydroxide	10 grams.
Water	100 c.c.

Loskiewicz reports that the nearly boiling solution colours cementite in 3–15 minutes. Over-etching causes deep pitting. There does not seem to be any great difference between Murakami's and Daeves' reagents as regards their action on iron carbide.

Alkaline Potassium Permanganate.

Potassium permanganate	4 grams.
Sodium hydroxide	1 gram.
Water	100 c.c.

According to Groesbeck (3) the above solution, used nearly boiling, has an etching action which almost coincides with Murakami's and Daeves' reagents. Possibly somewhat sharper definition is obtained with the permanganate. The etching time for cementite is 5–10 minutes. More strongly alkaline solutions, e.g. with 4 grams NaOH, reduce the time to 3–5 minutes.

Alkaline Potassium Copper Cyanide. Thompson and Whitehead found that alkaline potassium copper cyanide blackens cementite. A solution is prepared by adding 10 per cent potassium cyanide solution to 10 per cent copper nitrate until the precipitate first formed changes its colour to yellowish-green, with evolution of cyanogen (Note. Danger!), and is finally redissolved. The etching reagent is made from this solution by adding an equal volume of 50 per cent caustic soda.

With the hot reagent iron carbide is coloured in 1–5 minutes. Groesbeck (3) finds that this etchant yields better results than the alkaline picrate solution in general use; both lamellar pearlite, and spheroidized, as well as free, cementite are distinctly coloured by it. Even finely divided sorbitic cementite is blackened, while the ground-mass remains light and uncoloured.

The only disadvantage is the poisonous nature of the reagent.

Electrolytic Etching. According to Groesbeck (3) iron carbide is not attacked by electrolytic etching in either 10 per cent soda solution or 10 per cent caustic soda, thus distinguishing it from other carbides occurring in steels (*cf.* Table V, page 111).

Heat Tinting. Groesbeck (3) states that on tempering a polished surface at about 300° C. for 30 minutes cementite acquires a reddish-brown tint, while other carbides remain uncoloured. The ground-mass turns brown to blue, depending on the composition of the steel.

ETCHING OF HARDENED, AND OF HARDENED AND TEMPERED STEELS

A 1 per cent solution of hydrochloric acid in ethyl alcohol was proposed by Martens and Heyn in 1904 as an etching reagent for hardened steels. It may be used with advantage for steels merely hardened, or hardened and then tempered at a low temperature. According to the A.S.S.T. *Handbook,* 1 volume of the above solution can be diluted with 5 vols. distilled water and used for electrolytic etching with a weak current.

Hydrochloric Acid and Nitrophenol.

Solution A .	Saturated solution of orthonitrophenol in ethyl alcohol.
Solution B .	20% conc., HCl in amyl alcohol.

Kourbatoff recommends a mixture of 1 vol. A with 2 vols. B. With this reagent martensite needles and troosto-sorbite are coloured.

Nitric Acid in Ethyl Alcohol. Nitric acid in various concentrations in ethyl alcohol is probably the most widely used etching reagent both for hardened and unhardened steels. The relation of the etching action to the concentration of the reagent, and the time of etching is evident from Fig. 17 (after Loskiewicz). Sorbite and troostite are attacked considerably more rapidly than martensite. Therefore, in steels containing these constituents together, the two former will be very much over-etched before the structure of the martensite appears.

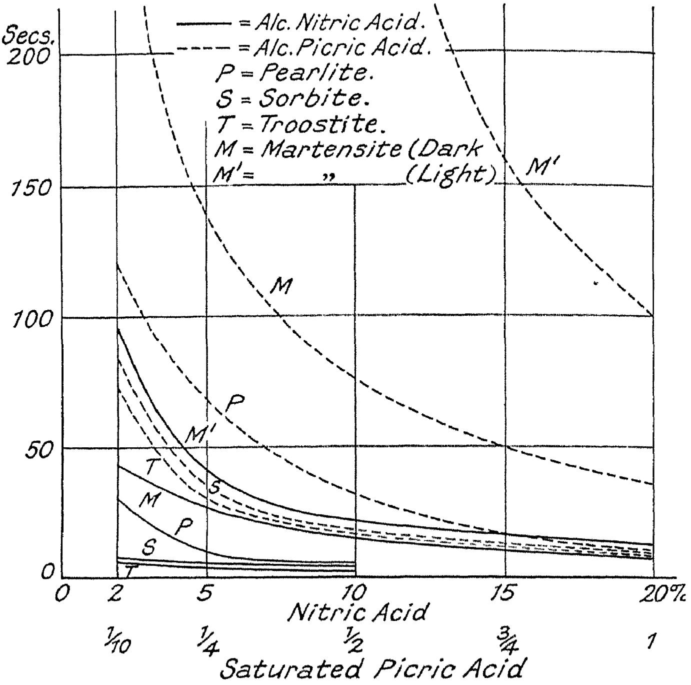

FIG. 17. ETCHING TIMES, AT 20° C., FOR PEARLITE, SORBITE, TROOSTITE, AND MARTENSITE IN 0·90 PER CENT CARBON STEEL (Loskiewicz)

The etching effect of nitric acid on hardened steel depends to a large extent on the formation of a relatively loosely adhering, coloured surface film. The various structural constituents react at different speeds, hence colours varying from pale yellow to brown and black are obtained, according to the time of etching.

When the colour as a whole begins to appear dark brown the surface layer is, in general, of such a thickness that the details of the structure are almost lost. Since the coloured film adheres quite loosely to the surface of the specimen, the latter must not be wiped after etching but dried freely, e.g. with an air-blast, after washing in hot water and alcohol.

After such etching the martensite needles appear dark against a lighter background. By wiping the surface under water after etching, or even by a brief and careful hand-polishing on a moist cloth, the martensite can be made to appear light against a darker background. This method, however, requires about a 50 per cent longer time of etching than when the specimen is not wiped. As a rule it is best not to use it when the specimen contains martensite and sorbite or troostite simultaneously.

Owing to the great difference in the etching times of martensite and troosto-sorbite, nitric acid dissolved in ordinary alcohol is, as previously pointed out, not very suitable as an etching reagent when these constituents are found together in one and the same specimen.

Boylston[1] particularly recommends 1 per cent nitric acid in absolute ethyl alcohol for etching steels containing martensite or troostite.

Nitric Acid in Amyl Alcohol. For hardened and tempered steel, and also for steels containing martensite and sorbite or troostite together, the 4 per cent solution of nitric acid in amyl alcohol, introduced by Kourbatoff, is considerably more suitable than solutions in ethyl alcohol. After 5 minutes etching, troostite and sorbite appear brown to black, or frequently greenish, while austenite is yellow, and martensite remains uncoloured.

Nitric Acid in Glycerine. Kourbatoff has prepared a 4 per cent solution of nitric acid in glycerine for distinguishing between austenite and martensite.

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[1] *A.S.S.T. Handbook.*

Hydrochloric Acid + Acetic Anhydride in Mixture of Alcohols. Kourbatoff recommends the following for etching sorbite and troostite—

Solution A	2 vol. methyl alcohol. 2 vol. ethyl alcohol. ol. iso-amyl alcohol. ol. butyl alcohol.
Solution B	4% HNO_3 in acetic anhydride.

7 vol. of Solution A are mixed with 3 vol. of B immediately before use. With up to 10 minutes etching this reagent only colours sorbite and troostite.

A modification of the above is—

Solution A .	1 vol. iso-amyl alcohol. 1 vol. ethyl alcohol. 1 vol. methyl alcohol.
Solution B .	4% HNO_3 in acetic anhydride.

3 vol. of Solution A are mixed with 1 vol. of Solution B immediately before etching. Colours troostite and sorbo-troostite.

Nitric Acid and Nitrophenol. According to Kourbatoff the various micro-constituents of hardened steel are well differentiated by the following reagent.

Solution A .	4% HNO_3 in ethyl alcohol.
Solution B .	Saturated solution of ortho-nitrophenol in ethyl alcohol.

1 vol. of A is mixed with 3 vol. of B.

Nitric Acid and Nitraniline. Given in the A.S.S.T. *Handbook.*

Solution A	4% HNO_3 in ethyl alcohol.
Solution B	Saturated solution of nitraniline in alcohol.

Mix 1 vol. of A with 3 vol. of B.

Picric Acid. 1 per cent picric acid in ethyl alcohol is recommended by Greene for etching sorbitic and troostitic steels. With this reagent the troostite appears lighter, and has a clearer structure, than after etching with 2 per cent HNO_3 in alcohol. The sorbite also develops a clearer structure than after using other etchants. Similarly the structure of the martensite is brought out.

According to Archer, and also Pulsifer and Greene, it appears to be possible to produce grain-boundary etching, to some extent, in hardened steel by a long etch with 4–5 per cent picric acid in alcohol. After etching, the surface film formed is removed by careful polishing by hand on a moist cloth. Probably the best result will be obtained by gently rubbing the specimen for the whole of the etching period so that no film has the opportunity of forming.

Picric Acid and Nitric Acid. Kourbatoff recommends a preliminary etching for 2 seconds in 4 per cent alcoholic picric acid, followed by 10 seconds in 5 per cent HNO_3 in isoamyl alcohol. Troosto-sorbite is coloured brown, and the heterogeneity of troostite is easily observed. Martensite acquires a bluish tint, and austenite turns orange.

The same writer states that an equally good etch is obtained with a mixture of 10 volumes of 4 per cent HNO_3 in methyl alcohol and 1 volume of 4 per cent picric acid in ethyl alcohol. Ten minutes etching colours sorbite, troostite, and the needles of martensite.

Metanitrobenzol Sulphonic Acid. Proposed by Benedicks (1) as a 5 per cent solution in ethyl alcohol. Colours martensite more strongly than austenite.

The latter is coloured brown by this reagent. With picric acid, of course, this is not the case (*cf.* pages 91 and 116).

Sulphurous Acid. According to Hilpert and Colver-Glauert a 3–4 per cent solution in alcohol or water is prepared as, and when, required from a saturated stock solution of SO_2 (about 10 per cent) in water. For the aqueous solution the time ot etching is 7 seconds to 1 minute, depending on the material, and several minutes in the case of the solution in alcohol. The specimen is immersed in the liquid with the polished surface upwards and, when the latter appears matt, it is taken out, washed in water and alcohol, and dried in an air-blast.

The etching effect is somewhat similar to that obtained by heat-tinting except that, instead of an oxide film, a thin layer of iron sulphide is formed. This reagent is recommended for hardened steels when it is desired to etch up the austenitic polygonal structure together with the martensite, the latter acquiring a pale blue colour and showing its needle structure. Manganese sulphide is stained dark blue by this etchant. It can only be used with unhardened or incompletely hardened steels if these are of eutectoid or hypereutectoid composition because the presence of free ferrite causes local pitting.

Ferric Chloride. Ischewsky mentions a solution of 5 per cent of ferric chloride in alcohol for etching hardened steels, but points out that the action is very strong, even more so than with iodine solutions.

Carbide Etching. The use of alkaline sodium picrate has already been described under normal steels (*cf.* page 92).

Nitric Acid in Methyl Alcohol and Nitrobenzol. A fuller description of this etching reagent will be found on page 114. It is recommended by Pilling for hardened steels because, although scarcely touching austenite, martensite, ferrite, or silico-ferrite, it attacks free carbide satisfactorily. Troostite is darkened, though not so much as with the usual acid reagents. Martensite is attacked very slowly.

It is very suitable for determining the presence or absence of free carbide, though in a few cases, it may happen that the martensite is also blackened by the reagent.

THE ETCHING OF CONSTRUCTIONAL ALLOY STEELS AND IRONS

The low-alloy special steels do not differ essentially in their etching properties from the ordinary carbon steels. For etching them the same reagents as for the latter steels are generally used (*cf.* page 87). As a rule, however, the time of etching has to be increased as the percentage of alloying element rises.

Structure Etching. "Hütte" states that 4 per cent picric acid in ethyl alcohol is often more suitable than nitric acid for etching special steels. Sometimes it may be an advantage to put 2 drops conc. HCl in every 25 c.c. of the picric acid solution.

Kalling recommends nitric acid in ethyl alcohol for etching low-chromium steels.

According to Pulsifer and Greene, the grain-boundaries of the austenite in hardened nickel-chrome steels can be etched up by Archer's method (alternate polishing and etching) with a 4 per cent solution of picric acid in ethyl alcohol, followed by the removal of the carbon-rich layer which is formed on the surface. Usually several minutes are required for etching, but the process must be watched continuously because over-etching of only 30 seconds results in an indistinct structure and numerous etch-pits. After etching the specimen is washed in alcohol, and kept moist with the latter while it is rubbed by hand on a polishing cloth. Finally, it is washed in hot water and dried in a powerful air-blast.

Carbide Etching. Westgren, Phragmén, and Negresco report that alkaline picrate does not always blacken the cementite of high chromium content found in ball-bearing steel.

Pilling's reagent (*cf.* page 114) is generally an excellent means of showing up free carbides in special steels.

HIGH CHROMIUM STEELS. STAINLESS AND HEAT-RESISTING STEELS

According to Kalling the choice of the etching reagent is of great importance when investigating high chromium steels. Thus, when using several of the common reagents, e.g. HCl, it has been found that austenite, and in certain cases even martensite, etches in very much the same way as ferrite, so that the distribution of the former is not clearly defined.

Hydrochloric Acid. Kalling and Pagels have used a 50 per cent solution of HCl in ethyl alcohol for etching chromium steels.

It is reported that a 10 per cent solution in alcohol can be used for stainless material.[1] The time of etching for the annealed steel is 3–4 minutes and, for the hardened, 20–30 minutes.

Hydrochloric Acid and Ferric Chloride. According to the A.S.S.T. *Handbook* the following solution can be used for etching stainless steels—

Ferric chloride	10 grams.
Hydrochloric acid, conc.	30 c.c.
Water	120 c.c.

The surface of the specimen is swabbed with cotton-wool saturated with the reagent. As a rule the etching time is not over 30 seconds. By diluting, a slower action is obtained. With a very dilute solution the etching can be performed electrolytically. This reagent was proposed by Curran.

The same authority also quotes a saturated solution of ferric chloride in hydrochloric acid, to which has been added a little nitric acid.

Hydrochloric Acid and Nitric Acid. The A.S.S.T. *Handbook* mentions "aqua regia" for etching stainless steels—

Hydrochloric acid, conc.	3 vol.
Nitric acid, conc.	1 vol.

The mixture should stand at room temperature for 24 hours before using. It etches extremely rapidly, and therefore requires careful handling.

Parmiter recommends the following mixture, which is used at ordinary temperatures—

Hydrochloric acid (sp. gr. 1·16)	4 parts
Nitric acid (sp. gr. 1·42)	3 parts
Water	4 parts

According to Vilella (1) the best etching reagent for iron-chromium alloys, low or high in chromium, is "aqua regia" in glycerine.

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[1] Communication from a Swedish works.

Nitric acid	1 vol.
Hydrochloric acid	2 vol.
Glycerine	3 vol.

In preparing the solution the acids are added separately to the glycerine. The reagent should be freshly made, as it becomes useless as soon as it acquires a strong colour of chlorine.[1]

For a specimen with low carbon content, repeated polishing and etching must be carried out to bring up the true structure. A time of about 30 seconds is used for the first etch. This etching reagent brings out the elaborate details of the structure better than any other reagent known at present, and Vilella specially recommends it for austenitic steels. Just before etching, the specimen is warmed slightly in hot water. The reagent is also applicable to iron-chromium alloys containing Al, W, V, or Mo. A modification of the above solution, more suitable for iron-chromium alloys low in carbon, and for partially austenitic alloys, is given by the A.S.S.T. *Handbook* as follows—

Nitric acid	1 vol.
Hydrochloric acid	3 vol.
Glycerine	2 vol.

The same authority quotes the following modification (by Vilella) of the reagent. This is well adapted for fully austenitic iron-chromium alloys, with or without other alloying elements such as Ni, Mn, etc.

Nitric acid	1 vol.
Hydrochloric acid	2 vol.
Glycerine	2 vol.
Hydrogen peroxide (3% ?)	1 vol.

The amount of hydrochloric acid can be altered according as the reagent is found to etch too quickly or too slowly. The method of alternate polishing and etching should be used to get the best result.

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[1] According to the writer's experience, some caution must be observed with glycerine solutions containing nitric acid because of the possibility of the formation of nitroglycerine.

Hydrochloric Acid and a Copper Salt. According to the A.S.S.T. *Handbook* the following reagent has been proposed by Marble for etching stainless material—

Copper sulphate	4 grams.
Hydrochloric acid	20 c.c.
Water	20 c.c.

Kalling reports that the various structural constituents can be particularly clearly discriminated with a reagent composed of—

Cupric chloride	5 grams.
Hydrochloric acid	100 c.c.
Ethyl alcohol	100 c.c.
Water	100 c.c.

The most suitable amount of HCl varies somewhat according to the composition of the steel, and its treatment. Kalling states that this etchant can be used with advantage for all steels containing more than 5 per cent chromium.

The etching action appears to be that ferrite is attacked and copper deposited upon it while the carbide remains unaltered. Austenite usually remains after etching as light as the carbide, but martensite always etches darker than ferrite. Hence, if the austenite has undergone a partial transformation into martensite it may acquire approximately the same colour as ferrite, and, therefore, be rather difficult to distinguish from the latter when occurring with it. In such cases it is generally possible to develop an increased colour contrast between the two phases by slightly changing the copper content of the etching solution. With Kalling's reagent it is possible to detect the merest traces of austenite.

Hydrochloric Acid and Mercurous Nitrate. The A.S.S.T. *Handbook* mentions the following reagent for etching stainless steels—

Hydrochloric acid	100 parts
Mercurous nitrate	7 parts
Water	100 parts

It is warmed to effect solution but used cold.

Electrolytic Etching. According to the A.S.S.T. *Handbook* a 0·5 per cent caustic soda solution is occasionally useful for the electrolytic etching of high chromium steels.

As also mentioned in the above, the process can be carried out in a dilute solution of ferric chloride and hydrochloric acid (Curran).

Furthermore, reference should be made to Velguth's electrolytic etching method for nickel-chrome described on page 142; in all probability this method would give successful results with chromium steels as well, but information on this point is lacking.

Carbide Etching. Groesbeck (3) states that chromium carbide is not coloured by either cold or boiling alkaline hydroxide solution after etching for up to 30 minutes. Concentrated solutions, however, discolour the entire surface of the specimen.

Guillet, Murakami, and Groesbeck (3) agree in reporting that alkaline picrate solution does not blacken chromium carbide.

Murakami's Carbide Etching Reagent. According to information received[1] this reagent is very useful for showing the carbides formed on annealing chromium and several other steels. As regards its composition, see page 93. The time of etching varies between 15 seconds and 3 minutes. Daeves reports that, at ordinary temperatures, the carbide phases in the eutectic in high-chromium carbon steels are also etched up.

Alkaline Potassium Permanganate. The reagent introduced by Groesbeck (*cf.* page 94) acts on chromium carbide in the same way as Murakami's reagent. In the boiling solution the time of etching is 1 minute.

Electrolytic Etching. According to Groesbeck (3) chromium carbide is attacked by electrolytic etching in 10 per cent soda solution as well as in sodium cyanide (see later, page 111).

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[1] Communication from a Swedish works.

HIGH NICKEL STEELS

For high nickel steels the etching reagents described for chromium steels can, in the majority of cases, be employed. Occasionally it is also possible to use those proposed for nickel itself (*cf.* page 140).

The A.S.S.T. *Handbook* quotes the following as an etching reagent for austenitic nickel steels—

Ferric chloride	5 grams.
Hydrochloric acid	50 c.c.
Water	100 c.c.

The practice at one Swedish steel works is to etch high nickel steels with 10 per cent ammonium persulphate. A 1 per cent solution of HCl in ethyl alcohol has been found useful for the grain-boundary etching of austenitic steels; slight relief-polishing is often an advantage.

According to Benedicks (2, 3) the presence of small amounts of the α-phase in "invar" alloys may be demonstrated by the method described on page 32, in which the surface of the specimen is first coated with a film of gelatine which is then hardened. After the treatment of the specimen in dilute acid, the iron-rich α-phase is "indicated" with a solution of potassium ferrocyanide which causes the appearance of local blue colorations in the gelatine film.

HIGH MANGANESE STEELS

Vilella states that the etching reagents containing glycerine devised for chromium steels (*cf.* page 103) are suitable for bringing out the structure of Hadfield's steels.

Belaieff shows that a good indication of the structure of hardened manganese steel can be obtained by etching first with 3 per cent nitric acid in ethyl alcohol, and then with Le Chatelier's No. 1 reagent (*cf.* page 57). The precipitated copper is dissolved off by treatment with strong ammonia. Belaieff states that the original dendritic cores of the austenite grains can be distinguished.

Sauveur and Krivobok report that the dendritic structure of cast manganese steel can be etched up, as a rule, by hot alkaline sodium picrate solution. This method, however, is considered to be hardly better than etching with some of the copper-containing macro-etching reagents, *e.g.* Stead's (*cf.* page 55).

Carbide Etching. For indicating carbides in manganese steels, Pilling's solution (*cf.* page 114) may be recommended, because it only etches the carbide network, and gives no sign of the distinctive banded appearance, due to manganese segregation, which is often brought out with other reagents.

HIGH SPEED AND OTHER HIGH TUNGSTEN STEELS

For annealed high-speed steel Gill and Bowman recommend structure etching with picric acid, and, for the hardened material, a 6–8 per cent solution of nitric acid in ethyl alcohol.

Amberg states that 5 per cent nitric acid in ethyl alcohol, with an etching time of about 10 minutes, is very suitable for grain-boundary etching. In the case of high-speed steel which has been hardened from a high temperature, and not subsequently tempered, only a polyhedral structure is obtained with the above reagent. In the hardened and tempered steel, however, a martensitic structure appears.

In those cases where only a polyhedral arrangement comes up on etching with nitric acid, Amberg has found that etching for half to one minute with the following solution often develops a martensitic structure—

Hydrochloric acid	20 c.c.
Water	15 c.c.
Ethyl alcohol	65 c.c.
Cupric chloride	1 gram.

The time of etching is increased as the proportion of hydrochloric acid is diminished.

Enlund[1] states that this reagent is neither more convenient nor better than HNO_3 in alcohol. He considers that Amberg's result was due to the specimen not being etched until some considerable time after polishing. Some martensite had meanwhile been formed in the surface layer of the metal, and it would, therefore, have appeared just as clearly if the etching had been carried out with nitric acid for example, instead of the above solution.

Hultgren reports that the structure of high-speed steel is brought out by "heat-tinting."

The A.S.S.T. *Handbook* states that Vilella's reagent for chromium steels (*cf.* page 103) can also be used for high-speed steels.

Carbide and Tungstide Etching. According to Groesbeck (3) iron tungstide is blackened in 1–5 minutes by cold 10 per cent alkaline hydroxide solution. The iron-tungsten-carbide in high-speed steel is blackened in about 10 minutes, but tungsten carbide is not darkened in up to 30 minutes etching.

The boiling solution blackens iron tungstide in 1 minute, but iron-tungsten-carbide is not blackened any more rapidly than in the cold solution. Tungsten carbide is not affected by the boiling reagent.

Alkaline Solution of Sodium Picrate. Hultgren states that iron tungstide is darkened more rapidly than iron carbide by the hot solution, while tungsten carbide is not so affected. This is confirmed by Groesbeck (3). The latter finds that iron-tungsten-carbide is blackened in 1½ minutes, and iron tungstide in 2 minutes.

Murakami's Etching Reagent. According to Groesbeck (3) this reagent should be freshly prepared when etching tungsten steels (*cf.* page 93). A steel containing about 5 per cent Cr, 18 per cent W, and 0·6 per cent C is etched by the cold solution in 15–20 seconds, and the carbides and tungstides are coloured brown to black, while the matrix remains uncoloured.

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[1] Private communication.

Groesbeck also reports that, in the boiling solution, tungsten carbide is blackened in 30 seconds, and iron tungstide and iron-tungsten-carbide in 60 seconds.

The action of the alkaline potassium permanganate, proposed by Groesbeck (*cf.* page 94), is absolutely identical with that of Murakami's reagent.

It has also been reported that Daeves' solution (*cf.* page 94) used hot, gives a sharply defined colouring of the secondary cementite, and of the cementite lamellae of the pearlite, in tungsten steels.

Alkaline Potassium Copper Cyanide. Groesbeck (3) states that Thompson and Whitehead's solution (page 94), when hot, colours iron tungstide and iron-tungsten-carbide in ¼ to 3 minutes. Tungsten, chromium, and vanadium carbides are not coloured. Iron carbide is coloured, but only relatively slowly.

Yatsevitch's Etching Reagent. Yatsevitch has proposed the following reagent—

Hydrogen peroxide (3%)	10 c.c.
Sodium hydroxide (10% solution) . .	20 c.c.

With an etching time of 10–21 minutes, tungsten carbide in high-speed steel is attacked, but iron carbide is not. During etching the gas-bubbles which form should be removed from time to time by brushing.

Groesbeck (3) finds that iron tungstide in carbon-tungsten steels is blackened in 1 minute, and iron-tungsten-carbide in high-speed steels in 3–5 minutes.

Neutral Solution of Sodium Picrate. Neutral sodium picrate is prepared by adding a saturated aqueous solution of picric acid to a strong solution of caustic soda, whereupon a yellow precipitate of sodium picrate is obtained. This is filtered off and dried in the air; it should be noted that the dry picrate is a very easily detonated explosive, so great care should be taken in handling it. A 1 per cent solution in water is then made up, and neutralized with aqueous picric acid, using litmus as an indicator (*cf.* page 118).

According to Groesbeck (3) the neutral picrate solution seems to be the only available reagent for differentiating iron tungstide and iron-tungsten-carbide in high-speed steels. Thus, in the boiling solution, the iron tungstide is blackened in 1 minute, but the iron tungsten carbide is not darkened after 10 minutes etching.

During the etching a brown surface film is generally formed, but it may be removed, while washing the specimen, by careful swabbing with very dilute hydrochloric acid.

Electrolytic Etching. Groesbeck (3) reports that tungsten carbide is attacked by electrolytic etching in 10 per cent soda solution, while iron tungstide and iron-tungsten-carbide are not affected. The two latter constituents are, on the other hand, attacked by electrolysis in 10 per cent caustic soda solution. See Table V.

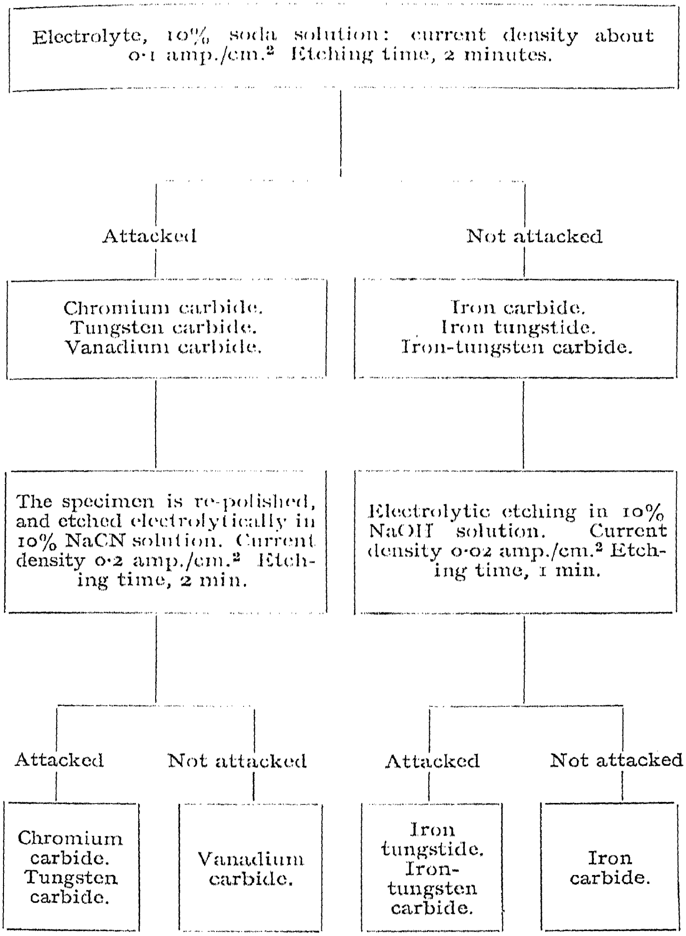
" Sequence Etching " for Identifying Structural Constituents in Tungsten Steels. According to Groesbeck (2) the most reliable information about the structure, particularly of material in the cast condition, is obtained by first etching with 5 per cent nitric acid in ethyl alcohol, whereupon the general structure is developed. Then, without intermediate polishing, the specimen is etched with Yatsevitch's reagent, so that any iron tungstide present is blackened. Finally, by etching with hot alkaline picrate solution, the iron carbide is similarly coloured. Thus, after this last process, the two blackened carbides can be easily observed, particularly in the cast material, because they usually form a network at the grain boundaries.

According to the Bureau of Standards (see Anon (3)) the specimen may be etched without intermediate

TABLE V

IDENTIFICATION OF CARBIDES OCCURRING IN STEELS BY ELECTROLYTIC ETCHING (GROESBECK)

Specimen positive electrode, platinum negative electrode.



Electrolyte, 10% soda solution: current density about 0·1 amp./cm.^{2} Etching time, 2 minutes.

Attacked

Chromium carbide.
Tungsten carbide.
Vanadium carbide.

The specimen is re-polished, and etched electrolytically in 10% NaCN solution. Current density 0·2 amp./cm.^{2} Etching time, 2 min.

Attacked

Chromium carbide.
Tungsten carbide.

Not attacked

Vanadium carbide.

Not attacked

Iron carbide.
Iron tungstide.
Iron-tungsten carbide.

Electrolytic etching in 10% NaOH solution. Current density 0·02 amp./cm.^{2} Etching time, 1 min.

Attacked

Iron tungstide.
Iron-tungsten carbide.

Not attacked

Iron carbide.

polishings in the following solutions successively, with microscopic examination after each etching.

1. Dilute solution of ammonia. (Electrolytic etch with specimen as anode.)
2. 2 per cent nitric acid in alcohol
3. Boiling solution of sodium picrate.
4. Murakami's reagent, hot.

VANADIUM STEELS

As regards general structure etching the vanadium steels do not differ from the corresponding carbon and alloy steels.

Carbide Etching. Vanadium carbide is not attacked by either cold or hot alkaline hydrate solutions; neither is it coloured by up to 15 minutes etching in hot alkaline picrate (Groesbeck (3)).

Potassium Ferricyanide in Faintly Alkaline Solution. According to conclusions which can be drawn from experiments carried out by Groesbeck (3), a modified form of Murakami's reagent should provide a means of differentiating vanadium carbide from cementite in steels free from other alloying elements. A solution of—

Potassium ferricyanide	10 grams.
Sodium hydroxide .	0·8 gram.
Water	100 c.c.

when used boiling, colours vanadium carbide in 10 minutes, while cementite is not attacked. It should, however, be noted that, with this reagent, both chromium and tungsten carbides, as well as iron tungstide, are blackened considerably more rapidly than the vanadium carbide.

Differentiating Chromium and Vanadium Carbides. Groesbeck (3) states that chromium and vanadium carbides can be distinguished by etching in a hot alkaline solution of potassium permanganate. One minute's etching colours the chromium carbide a dark reddish-brown or, more probably, dissolves out the carbide and colours the walls of the resulting cavities. The vanadium carbide does not appear to be attacked, and remains unchanged.

Another, but less satisfactory, method of differentiating the two carbides is by electrolysis in a weak solution of ammonium or sodium hydroxide in water. The chromium carbide is dissolved out leaving holes with the same reddish-brown appearance mentioned above, while the vanadium carbide is dissolved more slowly, leaving uncoloured cavities.

Vanadium carbide is not attacked by electrolytic etching in 10 per cent sodium cyanide, thus enabling it to be distinguished from chromium carbide and tungsten carbide (*cf.* Table V, page 111).

SILICON STEEL

According to Pilling, material containing silicon is attacked by water during polishing. The intensity of the attack increases with the content of silicon up to about 6 per cent of the latter, but subsequently falls off again with still higher percentages. For this reason alcohol should be used instead of water towards the end of the polishing process. Furthermore all etching reagents containing water should be avoided.

Structure Etching. Corson recommends the following etching reagent—

Glycerine . .	2 parts
Hydrochloric acid (30%)	2 parts
Nitric acid, conc. .	1 part

When etching a material containing 4 per cent of silicon a peculiar striated formation often appears in the structure. Corson states that this can be avoided to some extent if the material is previously annealed at 950° C., and cooled in air.

For high-silicon iron alloys Phragmén recommends etching with a mixture of 1 part conc. HNO_3 and 1 part HF (40 per cent). For low silicon materials this reagent is diluted with 1–2 parts of water. Time of etching, about half a minute.

Transformer steels, with about 3·5 per cent Si, are often etched with 10 per cent HNO_3 in ethyl alcohol, but as this easily roughens the surface of the ferrite, it is preferable to use Corson's reagent.

Baraduc-Muller etched iron alloys containing silicon with the two following reagents—

Hydrofluoric acid, conc.	1 vol.
Ethyl alcohol (95%) .	2 vol.
Distilled water .	1 vol.

Etching time, 2 minutes to 6 hours. If no action can be observed after 5 minutes, water is added drop by drop until the etching begins to be noticeable.

Nitric acid (sp. gr. 1·33) . .	100 vol.
Hydrochloric acid (sp. gr. 1·18) .	150 vol.

This mixture is warmed carefully until it turns brown. To 50 c.c. are then added 100 c.c. of a 47 per cent solution of ferric chloride. The action of the etching reagent can be diminished by the addition of water, say, 50 c.c. During etching a layer of SiO_2 often forms on the surface of the specimen, but it may be removed by treatment with a warm 10 per cent solution of ammonium fluoride.

Carbide Etching. Pilling points out that an alkaline solution of sodium picrate blackens ferrite containing silicon, and, therefore, is useless for indicating carbides in silicon iron and steels. He recommends the following reagent instead—

Stock solution	20% by weight, or 12·5% by volume, of fuming nitric acid in *absolute* methyl alcohol.

The methyl alcohol can be obtained free from water by distillation over quicklime. Forty drops of the stock solution are added to 50 c.c. of nitrobenzol. The reagent thus prepared is stored in a dark bottle, where it will keep for several months.

During etching, a film of organic iron compounds is formed but, after washing the specimen in alcohol, it may be removed by swabbing with strong caustic soda solution. Afterwards the specimen is again washed with water or alcohol, depending on the silicon content. A suitable time for etching is, generally, about 20 seconds. With this reagent only free cementite is coloured, while the silico-ferrite is left clear. It can also be used with advantage instead of hot picrate solution for showing free carbide in other steels besides those containing silicon.

According to the writer's experience, the action of the etching reagent depends entirely upon the methyl alcohol being free from water. The ordinary commercial alcohol must be redistilled over lime.

ETCHING OF CAST-IRON

The following is mainly as given by Bolton.

Picric Acid. A solution of picric acid is the best general etching reagent for grey cast-iron. Bolton uses a solution of—

Picric acid (saturated solution in alcohol) .	78 vol.
Nitric acid	2 vol.
Water	20 vol.

This acts uniformly and quickly, bringing out clearly the structure of the ferrite, pearlite, and steadite; 4–5 seconds etching is sufficient for examination at the higher magnifications.

The graphite in grey cast-iron easily absorbs some of the etching reagent, and, therefore, care must be taken to remove the moisture which the specimen usually exudes after drying, otherwise localized staining will occur.

Alkaline Sodium Picrate. Darkens cementite in the usual way. Ternary steadite (Fe_3P-Fe-Fe_3C) is also blackened, and the binary steadite (Fe_3P-Fe) is somewhat discoloured. Hence the solution cannot be used for differentiating Fe_3C and Fe_3P (*cf.* page 118).

Ammonium Persulphate. 10 per cent ammonium persulphate attacks both ferrite and pearlite, but leaves steadite white and unattacked. It is useful for distinguishing small areas of ferrite from phosphide at low magnifications.

Nitric Acid. Nitric acid in alcohol has essentially the same etching action as picric acid, but is seldom used for grey cast-iron because it causes considerable roughening of the surface. Campbell, however, recommends light etching in a 2 per cent solution for certain grey irons high in silicon in order to distinguish between pearlite and silico-ferrite.

Metanitrobenzol Sulphonic Acid. De Reyna and Daernis (*cf.* page 91) use a 5 per cent solution in ethyl alcohol for etching white irons, e.g. chill cast materials.

Phosphoric Acid. To demonstrate the presence of sulphur in the graphite lamellae, Bolton uses the following method.

The polished surface of the specimen is painted over with a thin layer of phosphoric acid (2 c.c. syrupy phosphoric acid + 90 c.c. water), and observed under the microscope with a 16 mm. objective. Bubbles of H_2S are evolved from the graphite if sulphur is present, but the metal itself is not attacked by the acid for some considerable time. (The correctness of this test is open to considerable doubt.)

Relief Polishing. This can be used with advantage for showing up steadite.

Heat Tinting. Bolton recommends a few seconds treatment of the specimen with 2 per cent phosphoric acid before heat-tinting.

The colours obtained on grey cast-irons are collected in tabular form opposite. It is an advantage to use the process for bringing out the net-structure of the graphite.

TABLE VI

HEAT TINTS ON GREY CAST-IRON

Colour of Specimen to Unaided Eye	Colour of Microconstituent at Magnification Stated
First perceptible yellow	*Steadite*
	× 100 White
	× 500 White matrix. Dark dots
	Graphite
	× 100 Black, lustreless. Purple edges
	× 500 Many blue dots along the edges
	Ferrite
	× 500 Brown with blue dots
	Pearlite
	× 500 Cementite: white. Ferrite: brown
	Average Colour
	× 100 Brownish yellow
Mainly brown, with no trace of purple	*Steadite*
	× 100 Pale greyish
	× 500 Greyish matrix with purple dots
	Graphite
	× 500 Red; purple and white along the edges
	Ferrite
	× 500 Brown to purple, with blue dots
	Pearlite
	× 100 Brown
	Average Colour
	× 100 Light brown
Dark brown changing to purple	*Steadite*
	× 100 The eutectic shows dark spots
	× 500 Blue dots. The dendrites have purple edges
	Graphite
	More blue and less purple along the edges
	Ferrite
	Like graphite
	Pearlite
	Purple and brown. The lamellae cannot be distinguished
	Average Colour
	Brown, with purple shades
Purplish red with blue spots	*Steadite*
	× 100 Purple edges
	× 500 Matrix yellowish-red
	Graphite
	As before
	Ferrite
	Blue and white
	Pearlite
	Red and purple
	Average Colour
	Red and blue

TABLE XI (*contd.*)

Colour of Specimen to Unaided Eye	Colour of Microconstituent at Magnification Stated
Blue and white	*Steadite* 500 Red matrix with white dots *Graphite* White and blue edges. Network structure appears at high magnification *Ferrite* White and blue *Pearlite* Blue and purple *Average Colour* Blue and white

ETCHING METHODS FOR IDENTIFYING PHOSPHIDES AND NITRIDES, AND VARIOUS NON-METALLIC INCLUSIONS

Iron phosphide occurs normally in the free form only in cast-iron, where it is a constituent of the ternary iron-carbon-phosphorus eutectic, the so-called steadite. The latter often resembles ledeburite in appearance, but as a rule it has a somewhat finer structure and, therefore, with a little experience, the two can generally be differentiated after an ordinary etch with picric or nitric acid. Iron phosphide is blackened, like cementite, by alkaline sodium picrate, and consequently neither can be distinguished by this reagent.

Oberhoffer and Poensgen etch phosphoric grey cast-iron for 20 minutes in 20 per cent HNO_3 in alcohol. The phosphide eutectic is not attacked, but appears light against a dark background, so that its amount can be easily estimated.

Etching with Neutral Sodium Picrate. Matweieff found that cementite and phosphide could be distinguished by etching with a neutral solution of sodium picrate, which blackens the phosphide.

According to Jungbluth, however, a strictly neutral picrate solution cannot be obtained in practice by merely neutralizing the alkaline picrate (*cf.* page 109), but must be obtained by recrystallization of the solid salt. Sodium picrate is prepared first by precipitation from a saturated aqueous solution of picric acid by means of cold concentrated soda solution. The precipitate is filtered off, and washed with cold water; then it is dissolved in warm water and recrystallized three times, whereupon a perfectly neutral picrate is obtained. When handling the sodium picrate it should be remembered that it is a powerful and easily detonated explosive which must be treated with due care, and not dried by warming, for example. A boiling, fairly concentrated, aqueous solution of the salt is used for etching, The etching time necessary is about 1½ hours, although the *alkaline* picrate darkens steadite in as little as 3 minutes. The reagent is satisfactory for both grey and white cast-irons. For successful etching it should be freshly prepared as a solution only a few days old often gives poor results.

Heat Tinting. Stead (3) and Wust (2) have shown that, on tempering, iron phosphide colours more slowly than cementite. When the latter has acquired a salmon red tint the phosphide is hardly yellow (Tables I and II, pages 29 and 30).

Czochralski (1) recommends tinting at 280° C. for 15–20 minutes. Bolton found that the colour contrasts were increased if, before heat-tinting, the specimen was etched extremely lightly with a 2 per cent solution of phosphoric acid (*cf.* page 116). Jungbluth, however, finds that, when using this latter method, very considerable experience is needed to get good results.

Goerens and Dobbelstein lightly repolished the surface after heat-tinting, so that the thin oxide film which had formed on the surface of the cementite was removed, but that on the other constituents remained. An excellent contrast for photography was thus obtained.

Solid Solutions of Phosphorus in Iron. Phosphorus in solid solution in iron may be indicated by any of the copper reagents described for macro-etching (*cf.* page 49).

Oberhoffer's reagent, and Fry's No. 1 and No. 2 solutions, are probably most suitable for micro-etching.

Nitrides. According to Kirner the iron nitride formed during the nitriding of steel appears with a characteristic brownish-yellow colour on etching the steel with 5 per cent picric acid in alcohol. For this reason he has proposed the name "flavite" for this microconstituent.

Comstock[1] reports that free iron nitride can be distinguished from cementite by etching in a hot, freshly prepared, 1–4 per cent solution of potassium ferricyanide. With this the iron nitride is not coloured, but cementite is blackened and pearlite turns brown. The *Werkstoffhandbuch* recommends for the same purpose an alkaline solution comprising—

Potassium ferricyanide	3 grams.
Potassium hydrate .	10 grams.
Water . . .	100 c.c.

According to B. Strauss the presence of nitride is best shown by heat-tinting at 250–300° C. The structural constituents containing nitrides are coloured most rapidly. When the ferrite has acquired a pale blue, and the pearlite a dark blue, colour, the nitrides and areas containing them appear red.

Titanium cyano-nitride and zirconium nitride sometimes occur in titanium and zirconium steels respectively. These constituents, however, have all the characteristics of non-metallic inclusions (see Tables VII and VIII).

Sulphides (*cf.* Tables VII and VIII). Gelatine reagents are now used extensively for indicating constituents which contain sulphur. A gelatine solution acidified with, for example, dilute sulphuric acid and slightly warmed, is mixed with a metallic salt of such a nature that the sulphuretted hydrogen evolved by contact with the specimen precipitates a characteristically coloured sulphide. For this purpose McCance used silver nitrate, and Law employed lead or cadmium salts.

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[1] *A.S.S.T. Handbook*, 1929, p. 331.

Whiteley made use of "tartar emetic" (potassium antimonyl tartrate), and his solution is probably the best known of this type of reagent. Five grams of French gelatine are allowed to swell for 1 hour in 20 c.c. distilled water. 10–15 c.c. of glycerine are then added, and the whole warmed on a water-bath until the gelatine melts, when further additions are made of 1 c.c. of a 7 per cent solution of "tartar emetic," and 1–1½ c.c. of dilute sulphuric acid (1 : 3). When using the reagent a drop of the slightly warm solution is placed on a cover-glass which is then carefully laid in contact with the surface of the specimen. Any sulphide particles are quickly surrounded by orange-coloured rings of antimony sulphide.

The acidified solution decomposes after standing for a short time, and, therefore, no more should be prepared than the occasion demands.

Mahin and Botts used Whiteley's antimony solution but omitted the gelatine. The specimen was merely dipped into the solution, and then carefully washed and dried. The sulphide particles were then surrounded by a clear yellow ring.

Kunkele has considerably improved the previous method. His process is as follows: 5 grams of gelatine are allowed to swell in 20 c.c. distilled water for half an hour, and then dissolved by gentle heating on a water-bath. When solution is complete, 20 c.c. glycerine and 2 c.c. sulphuric acid (sp. gr. 1·21) are added. The solution is stirred, and mixed with 0·8 gram silver nitrate. A brown coloration which may appear in the solution at this stage can be disregarded. The specimen, which is kept at a temperature of 20–25° C., is covered by means of a glass rod with 1–2 drops of the solution which has been warmed up to 27–28° C., and should be of the consistency of honey. After a few seconds the whole surface of the specimen is covered with a black silver coating which, after washing away the gelatine with hot water, is rubbed off under water with the finger or a piece of cotton wool. The specimen is then dried with alcohol. After this treatment the sulphide inclusions can be observed as dark coloured particles, surrounded by a light ring if they are embedded in ferrite or pearlite. If they are in cementite, ledeburite, or steadite, however, this light ring is absent. The method is applicable to ordinary irons and steels as well as to cast-steel, cast-iron, and pig-iron.

Comstock and others find that sulphide inclusions are blackened by hot alkaline sodium picrate solution in 5 minutes. With more prolonged etching they are entirely dissolved out.

According to the last-named authority; sulphides are blackened after 3 seconds etching in a 2 per cent solution of hydrofluoric acid in alcohol. However, silicates are also blackened, but oxides are not attacked. For slag and annealing scale a somewhat stronger solution is required.

Hydrofluoric acid	8 c.c.
Water . .	42 c.c.
Ethyl alcohol .	50 c.c.

With this reagent the time of etching depends entirely on the composition of the material. In the case of basic steels the etching effect with hydrofluoric acid is occasionally irregular owing to a needle-like structure appearing near the slag inclusions. This phenomenon, however, has not been observed very often in acid steels.

Matweieff reports that a 0·2 per cent aqueous solution of oxalic acid darkens sulphide inclusions in 20–30 seconds.

For showing manganese sulphide Hilpert and Colver-Glauert use a 3–4 per cent solution in water or alcohol of saturated aqueous sulphurous acid. Etching time, from 7 seconds up to several minutes. In the case of pig-iron high in manganese and sulphur the structure of the material is well developed and, at the same time, the manganese sulphide is coloured dark blue, and rendered clearly visible.

For identifying sulphides H. Kjerrman first etches the polished surface for 1–1½ minutes with a 1 per cent solution of oxalic acid in water. FeS is strongly attacked, but the MnS only very slightly or not at all. Oxides are also not attacked. Then the specimen is etched for 5 minutes in 10 per cent aqueous chromic acid, whereby the MnS is strongly attacked, while FeS and oxides remain untouched. After both these etchings all the sulphides are found to be blackened or dissolved away, while the other constituents of the structure have not been attacked.

According to Whiteley the colour of the sulphides is altered by heat-tinting rapidly at a temperature between 200° and 300° C. When the specimen as a whole has acquired an orange tint, the sulphide is already purple. The test is very suitable for identifying any Fe-FeS eutectic which may possibly occur.

Oxides and Other Slag Inclusions. (*cf*. Table VII). There is only a small amount of information in the literature concerning the identification of oxide inclusions by etching.

According to Law, iron oxide and iron-manganese oxide compounds are reduced if the specimen is treated electrolytically as the cathode in a nearly boiling solution of 0·002 per cent ammonium chloride or alkali hydrate in distilled water. Sulphides and silicates remain unchanged.

TABLE VII

ETCHING REAGENTS FOR IDENTIFYING (THE PRINCIPAL) NON-METALLIC INCLUSIONS

Reagent and Treatment	Sulphides	Oxides	Silicates	Special
Untreated, polished specimen	FeS brown-yellow (Mn,Fe)S dove grey, darker with increasing Mn content; smooth surface (MnS) dark dove grey	FeO grey; often rough surface (Fe,Mn)O grey blue darker than (Mn,Fe)S (Mn,Fe)O dark brown (MnO coloured by MnO_2) Al_2O_3 small hard rounded grains, never elongated by rolling SiO_2 larger, dark (transparent) hard grains, never elongated by rolling	Fe-silicate, black, greasy appearance Mn-silicate, greenish, greasy appearance, smooth surface	Sand grains: angular outline; greasy appearance; high relief Ti-nitride: yellow, rounded grains Ti-cyano-nitride: pink cubic crystals Zr-nitride: small lemon-yellow, cubic crystals Fe_3P: brilliant white (similar to Fe_3C)
Sulphuric acid, 1 part + H_2O, 50 parts. Best applied with a capillary tube under the microscope	H_2S bubbles	No bubbles	No bubbles	—
Chromic acid, 10% H_2O-solution; etch 5 minutes	FeS not attacked (Mn,Fe)S attacked	Not attacked	Not attacked	—
Hydrofluoric acid, 20% H_2O-solution. 10 minutes attack	Darkened somewhat	Not attacked	Attacked & darkened	—
Picric acid, 1% H_2O-solution (similarly also for acetic, citric, tartaric, and oxalic acids). Alcoholic solution acts five times more slowly	FeS strongly darkened in 1 minute (Mn,Fe)S only slightly darkened in 45 sec.	Not attacked	Not attacked	—
Neutral sodium picrate, boiling	Darkened	Not attacked	Not attacked	Fe_3P blackened (Fe_3C *not* blackened)
Alkaline sodium picrate, boiling (not more than 10 minutes)	Darkened	FeO not attacked (Fe,Mn)O attacked slightly (Mn,Fe)O attacked more strongly[1]	Attacked slightly	ZrN blackened

Ammonium carbonate, 2% H_2O solution; used warm, 20 min.	—	—	—	CaO . Fe_2O_3 destroyed
Hydrogen, at 600° C.	Not attacked	FeO & (Fe,Mn)O reduced (Mn,Fe) O and MnO not reduced	Not attacked	—
Hydrogen, cathodic (2 grm. NaOH + 1,000 C.C. H_2O)	Not attacked	FeO completely reduced (Fe,Mn)O reduced (Mn,Fe)O not reduced	Not attacked	—
Water vapour at 600° C.	Not attacked	(Fe), FeO and (Fe,Mn)O oxidized	Not attacked	—
Heat tinting at a bright red heat	—	FeO acquires a matt surface Fe_3O_4 unchanged	—	Fe_3P pale yellow (Fe_3C salmon), after previous ordinary etching
Heat tinting after ordinary etching, till iron turns yellow	FeS dark blue MnS greyish	—	—	—
Ferric chloride, dilute $C_2H_5.OH$ solution, used after reduction with gaseous or cathodic hydrogen followed by repolishing	—	FeO faintly coloured (Fe,Mn)O deeply coloured	—	—
Silver nitrate, 3% H_2O-solution; boiling, with light repolishing. (Similarly with mercury or gold salt)	FeS, red-violet (Mn,Fe)S unchanged	—	—	—
Alternatives, in acid solution in gelatine under cover-glass—				
Antimony salt (tartar emetia)	Yellow-red ring	No colouring	No colouring	—
Lead salt	Black ring	No colouring	No colouring	—
Cadmium salt	Pale yellow ring	No colouring	No colouring	—
Silver salt	Black, small ring	No colouring	No colouring	—

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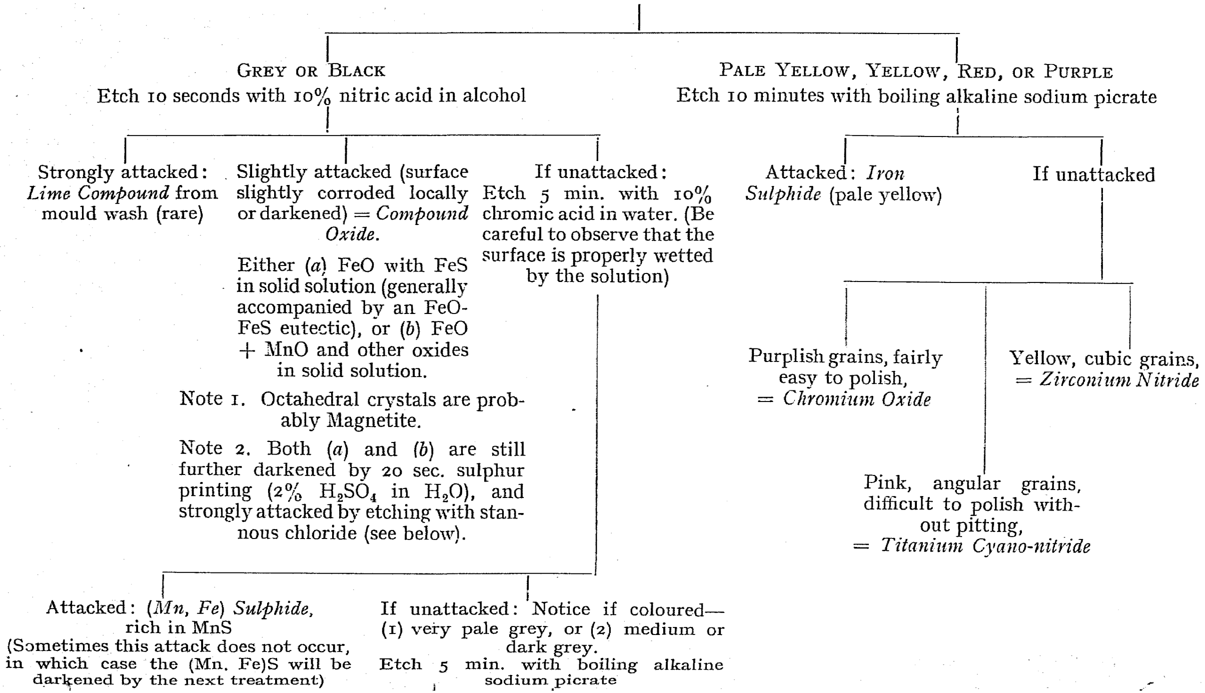
[1] Private communication from Mr. G. F. Comstock, and according to Wohrman.

TABLE VIII

SYSTEMATIC ANALYSIS

(FROM WOHRMAN)

Polish carefully so as to preserve inclusions and project magnified image on ground glass by arc or equally white light, without colour screen. Observe colours of inclusions present.

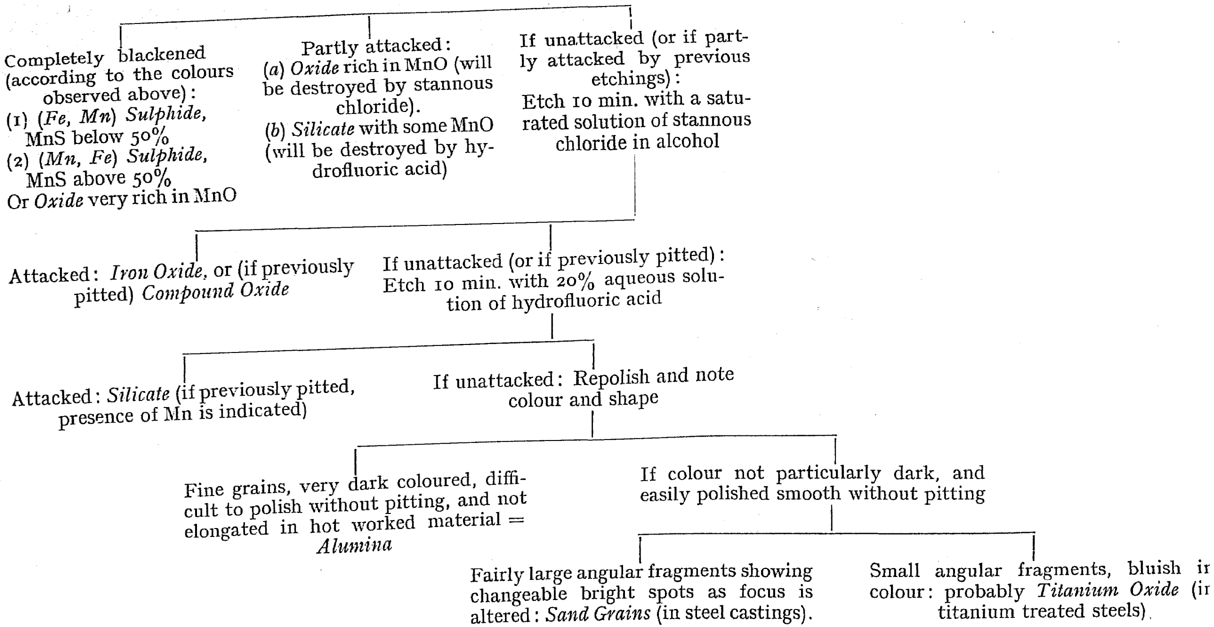


- GREY OR BLACK
  Etch 10 seconds with 10% nitric acid in alcohol
  - Strongly attacked: *Lime Compound* from mould wash (rare)
  - Slightly attacked (surface slightly corroded locally or darkened) = *Compound Oxide.*

    Either (*a*) FeO with FeS in solid solution (generally accompanied by an FeO-FeS eutectic), or (*b*) FeO + MnO and other oxides in solid solution.

    Note 1. Octahedral crystals are probably Magnetite.

    Note 2. Both (*a*) and (*b*) are still further darkened by 20 sec. sulphur printing (2% H_2SO_4 in H_2O), and strongly attacked by etching with stannous chloride (see below).
  - If unattacked: Etch 5 min. with 10% chromic acid in water. (Be careful to observe that the surface is properly wetted by the solution)
    - Attacked: (*Mn, Fe*) *Sulphide,* rich in MnS (Sometimes this attack does not occur, in which case the (Mn. Fe)S will be darkened by the next treatment)
    - If unattacked: Notice if coloured—(1) very pale grey, or (2) medium or dark grey. Etch 5 min. with boiling alkaline sodium picrate
- PALE YELLOW, YELLOW, RED, OR PURPLE
  Etch 10 minutes with boiling alkaline sodium picrate
  - Attacked: *Iron Sulphide* (pale yellow)
  - If unattacked
    - Purplish grains, fairly easy to polish, = *Chromium Oxide*
    - Pink, angular grains, difficult to polish without pitting, = *Titanium Cyano-nitride*
    - Yellow, cubic grains, = *Zirconium Nitride*



Completely blackened (according to the colours observed above):
(1) (*Fe, Mn*) *Sulphide*, MnS below 50%
(2) (*Mn, Fe*) *Sulphide*, MnS above 50%
Or *Oxide* very rich in MnO

Partly attacked:
(*a*) *Oxide* rich in MnO (will be destroyed by stannous chloride).
(*b*) *Silicate* with some MnO (will be destroyed by hydrofluoric acid)

If unattacked (or if partly attacked by previous etchings):
Etch 10 min. with a saturated solution of stannous chloride in alcohol

Attacked: *Iron Oxide*, or (if previously pitted) *Compound Oxide*

If unattacked (or if previously pitted):
Etch 10 min. with 20% aqueous solution of hydrofluoric acid

Attacked: *Silicate* (if previously pitted, presence of Mn is indicated)

If unattacked: Repolish and note colour and shape

Fine grains, very dark coloured, difficult to polish without pitting, and not elongated in hot worked material = *Alumina*

If colour not particularly dark, and easily polished smooth without pitting

Fairly large angular fragments showing changeable bright spots as focus is altered: *Sand Grains* (in steel castings).

Small angular fragments, bluish ir colour: probably *Titanium Oxide* (ir titanium treated steels).

SUPPLEMENTARY TESTS

Sulphur printing (for 20 seconds, using a 2% aqueous solution of H_2SO_4)—
(1) Dissolves all (Fe,Mn) sulphide inclusions.
(2) Partly dissolves (pits) FeO containing FeS and oxides of other metals in solid solution.
(3) Leaves unchanged iron sulphide (pale yellowish-brown), pure iron oxide (grey) and silicates (dark).

Campbell and Comstock state that iron and manganese oxide compounds are strongly attacked by a saturated solution of stannous chloride in alcohol.

Silicate inclusions are attacked by hydrofluoric acid solutions.

When examining finely divided slag, e.g. in cold rolled material, the inclusions usually appear more clearly if the polished surface is etched for 3–5 seconds in a solution of 2·5 grams iodine, 6 grams potassium iodide, and 1,000 c.c. water.[1]

For further information regarding the identification of slag inclusions, reference should be made to the recent book on *Non-Metallic Inclusions in Iron and Steel*, by C. Benedicks and H. Löfquist. (English translation published by Chapman and Hall, Ltd.) Tables VII and VIII on the preceding pages are taken, by permission, from this work.

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[1] Communication from a Swedish works.

CHAPTER V

ETCHING OF NON-FERROUS METALS

Copper. All etching reagents which are used for copper have an oxidizing action. Rawdon and Lorentz (1) carried out a critical examination of these reagents, and their results are summarized below.

Oxidizing Acids. (*a*) Concentrated nitric acid occasionally gives a useful etch, but the time of etching is extremely short and, therefore, difficult to control.

(*b*) Dilute nitric acid (1 part acid + 1 part water) attacks the copper very uniformly, but indistinct contrasts are obtained. (Etching time, 15–30 seconds).

Nitric acid has no advantages over other etchants for copper and, therefore, is seldom used. Solution (*b*) can, however, be recommended for preparing the specimen by Pulsier's method of repeated polishing and etching.

Chromic Acid. (*a*) Saturated solution. Time of etching 1–1½ minutes. Attacks the grain-boundaries and oxide globules particularly. Seems to be suitable for etching cold-worked copper.

(*b*) A 10–15 per cent solution, with the addition of 1–3 drops of HCl per 50 c.c. of solution is stated by Vilella (2) to be a satisfactory reagent. Gives a pronounced grain contrast etching. Alternate polishing and etching can be used with advantage.

Sulphuric Acid. (*a*) Sulphuric acid and hydrogen peroxide.

Sulphuric acid, conc.	1 part
Hydrogen peroxide (3%)	20 parts

Etching time, 1–1½ minutes.

(*b*) Sulphuric acid and potassium bichromate.

Sulphuric acid, conc. . . .	1 part
Potassium bichromate (sat., solution)	10 parts

Etching time, 30–60 seconds. Very suitable for cast copper, but attacks oxide inclusions strongly.

(*c*) Sulphuric acid and potassium permanganate.

Sulphuric acid, conc.	1 part
Potassium permanganate (0·4% solution)	10 parts

Etching time, 1–1½ minutes. Good general reagent for copper.

Hydrochloric Acid. (*a*) Hydrochloric acid and ferric chloride in aqueous solution.

Hydrochloric acid, conc.	30 c.c.
Ferric chloride.	10 grams.
Water	120 c.c.

Etching time, 30 seconds.

(*b*) Hydrochloric acid and ferric chloride in alcoholic solution.

Hydrochloric acid, conc.	30 c.c.
Ferric chloride.	10 grams.
Ethyl alcohol	120 c.c.

Etching time, 30 seconds.

(*a*) and (*b*) both give good grain-contrast etching for low magnification. For the higher powers, the surface is roughened too much. The alcoholic solution gives better contrasts than the other.

Ammonia Aided by the Oxidizing Action of the Atmosphere. According to Czochralski (3) the surface of the specimen is swabbed with cotton wool moistened with strong ammonia. The etching reagent is spread as evenly as possible over the surface, and any dark coloured patches which appear are removed by careful rubbing, so that a clear metallic surface is obtained. When the etching process is to be stopped a few drops of water are added to the wad of wool, without ceasing the swabbing, and then more and more water is successively added until all the ammonia has been washed away. The use of excess of ammonia delays the attack because oxidation is hindered. If too little is employed a layer of cellulose is deposited on the surface of the specimen, and it has to be removed with fresh ammonia. As a result of the above treatment the attack is mainly confined to the boundaries of the grains while, generally, their surfaces remain smooth.

Guertler (2) reports that ammonia may sometimes be used in the polishing of copper specimens.

Ammonia and Hydrogen Peroxide. (*a*) Equal parts of strong ammonia, and 3 per cent hydrogen peroxide. Etching time, 5–15 seconds. Gives principally a grain-boundary etch which is very suitable for investigations at high magnifications. Ammonia and hydrogen peroxide is one of the most commonly used etching reagents for copper. By varying the proportions of the ammonia and the peroxide the etching effect can also be changed to some extent. If the amount of peroxide is increased, a grain-contrast etching is obtained. The A.S.S.T. *Handbook* recommends, for cold-worked material, a subsequent etch for a few seconds with the solution (*a*) described under hydrochloric acid (*cf.* page 130).

(*b*) Grain-contrast etching is obtained with—

Ammonia, conc. .	1 part
Hydrogen peroxide (3%) .	20 parts

(*c*) For cast copper, swabbing with the following solution is recommended in the A.S.S.T. *Handbook*—

Ammonia, conc. . . .	40 c.c.
Hydrogen peroxide (3%) .	10 c.c.

This solution can also be used for "polish attack."

Ammonia and Potassium Permanganate.

Ammonia	2 parts
Potassium permanganate (0·4% solution)	3 parts

Etching time, 2–3 minutes. The solution etches best when it begins to acquire a brownish colour owing to the formation of manganese dioxide. Under other conditions a film easily forms on the surface of the specimen and masks the etching.

Ammonia and Weak Oxidizing Agents. (*a*) Ammonia and ammonium oxalate.

Ammonia, conc.	1 part
Ammonium oxalate (sat. solution)	3 parts

Ammonia and potassium arsenate.

Ammonia, conc.	1 part
Potassium arsenate (sat. solution)	3 parts

Both these reagents act very slowly. Time of etching, about 24 hours. Solution (*b*) attacks copper oxide globules strongly.

Ammoniacal Copper-ammonium Chloride Solution.

Copper-ammonium chloride	5 grams.
Water	120 c.c.

Strong ammonia is added till the precipitate first formed is redissolved. Etching time, 30–60 seconds.

This is one of the best, and most commonly used, general reagents for copper, giving chiefly grain-contrast etching. The alcoholic solution gives poor results.

Ammonium Persulphate. A 10 per cent solution of ammonium persulphate in water is an excellent etching medium for copper, and is often employed. Better results can sometimes be obtained if a few drops of ammonia are added immediately before etching. A pronounced relief-effect is obtained, depending on the crystallographic orientation of the grains.

Bromine Water. A saturated solution of bromine in water often gives a good etch in 30–60 seconds. The coating which forms on the surface of the specimen is washed off in strong ammonia.

Silver Nitrate. (*a*) 2 per cent solution of silver nitrate. Etching time, 20–30 seconds. The silver layer precipitated on the specimen is rubbed off under water. Oxide globules are strongly attacked.

(*b*) In Holman's method a small particle of dry silver nitrate is laid on the specimen and a single drop of water added. As a result there is formed a light spongy layer of silver which changes colour to grey in about 3 seconds. The surface is then immediately flushed clean with water from a wash-bottle. With correct technique a good etch is obtained. The method seems to be valuable in those cases where other reagents produce undesirable etch-pits.

Electrolytic Etching. The electrolytic etching of copper can be carried out in, for example, a 10 per cent solution of ammonium acetate, or 10 per cent ammonium sulphate. Current density about 0·003 amp/cm². Time about 30 minutes. As a rule, however, electrolytic etching gives a roughened surface which is, therefore, not suitable for observation at high magnifications. Any residual grinding scratches are strongly attacked.

COPPER ALLOYS

The single-phase copper-rich alloys, e.g. α-brass and α-bronze, have, in general, the same etching properties as pure copper, and, therefore, the same etching reagents can be used. As the copper content falls the oxidizing character of the reagent can with advantage be somewhat reduced.

In alloys containing several phases, the differences in the rates of solution of the latter will be increased owing to the setting up of local elements. Hence, a strong grain-contrast etching is obtained.

A method, which was used successfully by Rawdon and Lorentz for a number of copper alloys, consists of a double etching with sulphuretted hydrogen, and 10 per cent silver nitrate solution. Marked contrasts are obtained in this way. In some cases the best result is produced if the sulphuretted hydrogen treatment is carried out first, while in others it is preferable to begin by etching with the silver nitrate. A test with an alloy containing 67 per cent Cu, 32 per cent Zn, and 1 per cent Pb gave the best result when the specimen was first placed in a flask kept filled with H_2S, and with a drop of HCl in the bottom. The specimen should be left until a thin sulphide film has formed, and then etched in the 10 per cent silver nitrate. The method has been found to be satisfactory for α- and $\alpha\beta$-brass. As regards a bronze containing 88 per cent Cu, 10 per cent Sn, and 2 per cent Zn, the best result was obtained if the silver nitrate etch was carried out before the treatment with H_2S. The chemical nature of the film formed as a result of the action of the H_2S does not seem to be of paramount importance from the point of view of the method, because a similar result was obtained if the specimen was heated in air so that a thin oxide layer was formed.

According to Rawdon and Lorentz (3) the action of very weak etching reagents can often be accelerated by bubbling oxygen through the solution. Excellent results can be obtained in this way for many copper alloys when using strong ammonia, or dilute sulphuric acid, or a 10 per cent solution of ammonium chloride. The oxygen is passed in through a fine tube, and the stream is directed on to the specimen. As a rule, a comparatively long etching time is required. With the above treatment the specimen is not usually etched over its entire surface, but only in an annular zone surrounding the place where the stream of oxygen impinges upon it.

For the macroscopic etching of copper alloys Frémont recommends the following reagents—

(*a*)	Nitric acid	15 c.c.
	Hydrochloric acid	8 c.c.
	Water	100 c.c.
(*b*)	Nitric acid	67 c.c.
	Hydrochloric acid	33 c.c.
(*c*)	Concentrated hydrochloric acid.	
(*d*)	Sulphuric acid	2 vol.
	Water	10 vol.
(*e*)	10% solution of ferric chloride in water.	

Of these etching reagents, (*d*) has the slowest action.

Etching of α-Brass and α-Bronze. A group of etching reagents examined by Rawdon and Lorentz is given below, and the first four may be regarded as universal reagents for the majority of copper alloys. Nevertheless the action of any given reagent often varies according to the composition, mechanical treatment, and heat-treatment of the material. No general rules for these variations in etching properties can be laid down, however, and it is necessary to find out by trial the best conditions for etching in each case.

TABLE IX

	Etching Reagent	Etching Time	Character of Etching	Remarks
(*a*)	Ammonia, conc., 10 vol.. Hydrogen peroxide (3%), 1 vol.	15–30 sec.	Contrast	Swabbing often useful
(*b*)	10% ammonium persulphate	20 sec.–5 min.	Contrast	Alcoholic solution usually gives poorer results, but can occasionally yield better
(*c*)	Alkaline copper ammonium chloride (see under copper)	20 sec.–1 min.	Grain-boundary	
(*d*)	Saturated chromic acid solution	10–30 sec.	Contrast	
(*e*)	Sulphuric acid, conc., 1 vol Hydrogen peroxide (3%) 10 vol.	30 sec.–2 min.	Contrast	
(*f*)	Sulphuric acid, conc. 1 vol. $H_2Cr_2O_7$, sat. solution, 5 vol.	10 sec.–1½ min.	Variable	Suitable for low magnifications
(*g*)	Nitric acid, conc.	1–4 sec.	Contrast	
(*h*)	Hydrochloric acid, conc., 30 c.c. Ferric chloride, 10 gr. Water, 120 c.c.	20 sec.–1 min.	Contrast	
(*i*)	Hydrochloric acid, conc., 30 c.c. Ferric chloride, 10 gr. Alcohol, 120 c.c.	30 sec.–1½ min.	Contrast	Sometimes better and sometimes worse than aqueous solution
(*j*)	2% silver nitrate solution			

Etching of $\alpha\beta$-Brass and $\alpha\delta$-Bronze. In the case of brass, according to Rawdon and Lorentz, it is found that etching reagents with a powerful solvent action, e.g. strong nitric acid, or sulphuric acid + potassium bichromate solution (page 129), attack the β-phase strongly, so that this, in intaglio, appears dark against the sound-mass of the α which is attacked to a less degree. Usually grain-boundaries are etched up in the latter phase.

Reagents with a less powerful solvent action, e.g. sulphuric acid + hydrogen peroxide, sulphuric acid + potassium permanganate, ammonium persulphate, hydrochloric acid + ferric chloride, and bromine water, colour the β-grains, so that they appear dark against the lighter, structureless α ground-mass. When using an etching reagent which has a special affinity for copper, e.g. ammonia + hydrogen peroxide, ammonia + potassium permanganate, and ammoniacal copper ammonium chloride solution, the opposite effect is obtained, and the β-crystals appear light against the somewhat darker α-background.

According to Bauer and Hansen 10 per cent chromic acid colours the β-crystals a beautiful lemon-yellow, while the α-crystals are coloured ochre-yellow.

In alloys which are chiefly composed of the α-phase, a contrast etching is often obtained between the various α-crystals, and as a result any β-areas present do not stand out so distinctly.

Abnormal etching effects can sometimes be obtained as a result of the presence of segregates or of persistent ingot-structure; they occur more often with such reagents as ammoniacal copper ammonium chloride, or ferric chloride and sulphuric acid + an oxidizing agent, than with, for example, silver nitrate or chromic acid.

Bronze is, in general, analogous to brass in this respect.

According to the A.S.S.T. *Handbook*, ammonia + hydrogen peroxide is as good an etchant for brass as for bronze. After etching in this reagent the specimen is dipped into a ferric chloride solution, the concentration of which should be determined for each particular case.

Campbell reports that the dendritic structure in cast bronze shows up clearly after etching with 50 per cent nitric acid, but the eutectoid is so completely blackened that its structure is lost. On the other hand, swabbing with ammonia + hydrogen peroxide attacks the copper-rich cores of the dendrites, without affecting the eutectoid. The latter is, therefore, seen in very faint contrast against the saturated α-solution. If, however, the specimen is subsequently washed over with Grard's No. 1 solution—

Ferric chloride .	0·5 gram.
Hydrochloric acid, conc.	50 c.c.
Water . . .	100 c.c.

till the α-phase is slightly darkened, the eutectoid with its pale bluish-white colour remains in distinct contrast.

When etching with chromic acid Miller added hydrogen peroxide. Vilella (2) found, however, that the effect of the peroxide was, in this case, chiefly determined by the small percentage of hydrochloric acid which was added as a preservative, and that better etching was obtained with chromic acid + hydrochloric acid (*cf.* page 129). In $\alpha\beta$-brass the α-phase is not attacked, but the β is coloured light yellow as with persulphate. In copper-tin alloys there is no effect on the α-phase, but the δ is coloured brown to black, depending on the time of etching. In rolled alloys the grain boundaries and any "twinning" stand out clearly. Miller found that chromic acid was a good etching reagent for welds, particularly when manganese bronze was used, as, in this case, etching with ferric chloride was unsuccessful.

Uneven etching or staining easily occurs if the chromic acid is not pure, e.g. if it is contaminated with sulphuric acid. Similarly, care should be taken that no alcohol or other reducing agent gets into the solution, which should be freshly prepared, and also should be thrown away as soon as it becomes dark coloured. The specimen should be kept in motion while etching, and then immediately washed in hot water.

According to Vilella (2) this reagent is the only one which can be used to etch up the particles of the silicides of nickel, cobalt, chromium, or iron in certain copper alloys (Corson's alloys). The etching time is usually 5–10 seconds.

Copper-zinc Alloys, High in Zinc. Of the copper-zinc alloys containing high percentages of zinc, only the β-alloys have any great practical interest. For a cast alloy containing 53·9 per cent Cu and 45·7 per cent Zn, Rawdon and Lorentz (3) found that—

(*a*) good grain-contrast etching was obtained with—

Ammonia + hydrogen peroxide (10 : 1)	Etching time 15 sec.
Ammonia + potassium permanganate	,, ,, 1½ min.
Sulphuric acid + potassium bichromate	,, ,, 30 sec.

(*b*) good grain-boundary etching was obtained with—

Ammonia + ammonium oxalate or potassium arsenate	Etching time 20 sec.
Bromine water	,, ,, 20 sec.

A cast $\beta\gamma$-alloy containing 44·7 per cent Cu and 41·0 per cent Zn, gave pronounced contrast-etching, γ dark and β light, with ammonia + hydrogen peroxide, ammonia + potassium permanganate, hydrochloric acid + ferric chloride in alcohol, alkaline copper-ammonium chloride, and bromine water. With sulphuric acid + potassium bichromate the β etched dark. Mainly grain-boundary etching was obtained with sulphuric acid + hydrogen peroxide, ammonium persulphate, saturated chromic acid, and hydrochloric acid + ferric chloride in water.

Lead in Brass and Bronze. When lead is present it is best observed on polished, unetched surfaces. On etching lead-containing alloys with certain reagents, particularly ammonia + potassium permanganate, a light unetched area is obtained around each particle. Knight proposed etching with a 10 per cent solution of an alkaline sulphide, or treatment for about 5 seconds with sulphuretted hydrogen. According to Gilligan and Curran the same effect is obtained with ammonia + hydrogen peroxide, or with ferric chloride solution. The Bureau of Standards (2) reports that a double etching can be carried out with advantage, the first etch being with ammonia + hydrogen peroxide, followed by treatment with ferric chloride solution.

Bolton recommends nitric acid for etching brass and high-tin bronze when rich in lead.

Aluminium Bronze. In the "as-cast" condition aluminium bronze is etched without difficulty by the majority of the reagents employed for the various copper alloys, and, according to Rawdon and Lorentz, also by strong hydrochloric acid, the etching time being about 15 minutes.

In the worked condition, however, the material is occasionally rather troublesome to etch. J. Strauss recommends the use of magnesium oxide for polishing the specimen.

Up to the present, ferric chloride or various mixtures of nitric and chromic acids have been used for etching worked aluminium bronze, although the process is often hindered by the formation of some kind of surface film on the specimen. Strauss finds that, as a rule, this can be avoided by treating the specimen with a 10 per cent aqueous solution of hydrochloric acid, for 2–5 seconds, before the proper etch. After the hydrochloric acid treatment the specimen is washed in cold water, and then quickly immersed in the etching reagent to be used. Strauss tested the following reagents—

(*a*)	Nitric acid	50 c.c.
	Chromic acid	20 grams,
	Water	30 c.c.

(*b*)	Nitric acid	5 c.c.
	Chromic acid	20 grams.
	Water	75 c.c.
(*c*)	Hydrochloric acid	20 c.c.
	Ferric chloride.	10 grams.
	Water	120 c.c.
(*d*)	Nitric acid	30 c.c.
	Water	70 c.c.
(*e*)	Ammonia	10 c.c.
	Water	5 c.c.

All these reagents gave grain-boundary etching. The etching with ammonia resulted in slight grain-contrast but less definition of the twinned-structure.

Alloys with high percentages of iron and sufficient aluminium to form the β-solution, e.g. 8·8 per cent Al, 3·7 per cent Fe, and 87·5 per cent Cu, etch well with solution (*b*) quoted above. The zone-structure of the α-crystals appears distinctly, and this is not the case with many other reagents. The iron-component appears as blue-grey to dark brown particles, depending on the time of etching.

Etching in strong ammonia (sp. gr. 0·90) while passing oxygen into the solution, as described by Rawdon and Lorentz (3), gave very good results, but an etching period of about 30 minutes was required.

NICKEL AND NICKEL ALLOYS

Nickel is one of the most difficult metals to etch in a satisfactory manner. Owing to its great resistance to chemical action it is necessary to resort to strong etching reagents. With these, however, there is often the disadvantage that deep etchpits are formed around any non-metallic inclusions present, and this has a very disturbing effect on the development of the grain structure.

The etching of nickel is, in the majority of cases, a grain-boundary effect without any kind of contrast between the surfaces of the various grains. The difficulties of etching increase with the purity of the metal. Occasionally the specimen has to be re-polished and re-etched many times before a satisfactory result can be obtained.

Of the etching reagents for nickel given below, the first three should be regarded as the most generally useful.

Concentrated Hydrochloric Acid. According to Rawdon and Lorentz (2), strong hydrochloric acid is one of the best etching media for nickel. The etching time is, however, about 1 hour. The A.S.S.T. *Handbook* recommends this reagent specially for cast, and for cold-worked, nickel.

Nitric Acid + Acetic Acid. Merica (see Bureau of Standards (1)) recommends nitric acid + acetic acid as one of the best etching reagents.

Nitric acid (70%)	50 c.c.
Glacial acetic acid (50%)	50 c.c.

Merica's reagent has been modified subsequently by the International Nickel Co. by the addition of acetone. (A.S.S.T. *Handbook*).

Nitric acid	20–40 c.c.
Acetic acid (75%)	30–40 c.c.
Acetone	30–40 c.c.

Both these reagents can be used equally well either for cast, or for cold-worked and annealed nickel.

Ammonium Persulphate. An approximately 10 per cent solution of ammonium persulphate is often useful, especially for cast nickel.

Nitric Acid. According to the A.S.S.T. *Handbook*, concentrated nitric acid is suitable for electrolytic, and cold-worked nickel. Etching time about 5 seconds.

50 per cent nitric acid readily produces etch-pits surrounded by coloured rings. Generally, Merica's reagent is preferable.

Sulphuric Acid + Oxidizing Agent. According to Rawdon and Lorentz (3) the following reagents can sometimes be used for the etching of nickel.

Sulphuric acid, conc.	1 vol.
Hydrogen peroxide (3%)	10 vol.

Sulphuric acid, conc.	1 vol.
Potassium bichromate (sat. solution)	5 vol.

Hydrochloric Acid + Ferric Chloride. The Bureau of Standards (1) states that the following solutions give a satisfactory etch—

Hydrochloric acid	30 c.c.
Ferric chloride	10 grams.
Water	120 c.c.

Hydrochloric acid	2 c.c.
Ferric chloride	1 grm.
Water	2–3 c.c.

Aqua Regia. Vilella reports that a solution of aqua regia in glycerine is occasionally a useful etching reagent for nickel.

Nitric acid	1 vol.
Hydrochloric acid	3 vol.
Glycerine	2 vol.

According to "Hütte" a dilute solution is useful.

2% hydrochloric acid in alcohol	3 vol.
2% nitric acid in alcohol	2 vol.

Picric Acid + Hydrochloric Acid. "Hutte" gives—

Picric acid	1 gram.
Alcohol	25 c.c.
Hydrochloric acid (sp. gr. 1·12)	2 drops

Electrolytic Etching. According to the Bureau of Standards (1) nickel can be etched electrolytically in a solution of sulphuric acid and hydrogen peroxide—

Sulphuric acid	22 c.c.
Hydrogen peroxide	12 c.c.
Water	66 c.c.

Velguth reports that nickel-chrome can be etched satisfactorily by being treated first as the cathode in a 10 per cent sodium hyposulphite solution (or 10 per cent NaOH solution) for 5–10 seconds with a current-density of about 0·24 amp/cm.^{2}, at 10 volts (activating process). The specimen is then transferred without intermediate washing, and, therefore, with the surface covered by hyposulphite solution, to a bath of 10 per cent hydrochloric acid, and, at the same time the polarity of the specimen is changed over so that it now acts as the anode. It is electrolyzed in the acid for

8–10 seconds, with a current density of 0·1 amp/cm.^{2} To avoid rendering it passive the specimen must be electrically "alive" on immersion in the hydrochloric acid. This double electrolytic treatment is most easily accomplished by putting a reversing switch in the circuit together with a regulating resistance which has been previously marked in the correct positions for obtaining the desired strengths of current. The specimen is conveniently held with crucible tongs which are connected in the circuit, and the opposite electrode can be made of a piece of graphite or arc-lamp carbon.

Velguth's method, which has been found excellent for nickel-chrome, might in all probability be applicable to nickel as well.

Nickel Alloys. Nickel alloys are, throughout, considerably more easily etched than pure nickel. The alloys in commercial use are mainly of the α-type, i.e. single-phase solid solutions. In the cast form, however, they generally exhibit a marked dendritic structure, and, as a consequence, in the rolled state a fibre structure which in many ways is suggestive of that caused by phosphorus in iron and steel. Strongly oxidizing etching reagents bring out segregates.

Nickel-copper Alloys. Cupro-nickel, Monel metal, nickel silver, etc., can all be etched with the reagents described for nickel. When the nickel content is low most of the solutions for copper are also serviceable.

For cast Monel metal Campbell recommends Merica's reagent somewhat diluted. In rolled and annealed Monel metal Grard's solution gives grain-contrast etching (*cf.* page 137).

Norbury etched cupro-nickel by polish-attack on parchment with ammonia + ammonium persulphate.

Nickel-chromium Alloys. Vilella (1) states that nickel-chromium can be etched with a solution of aqua regia in glycerine.

According to the author's confirmatory tests, the electrolytic method of Velguth described above is greatly superior, and gives brilliant etchings on the nickel-chromium alloys used for resistance and heating purposes, as well as on wires for thermo-elements.

ALUMINIUM AND ALUMINIUM ALLOYS

For the macro-etching of aluminium and its alloys it is preferable to use a combination of hydrofluoric and hydrochloric acids.

According to Czochralski (1) the specimen is first etched for some time in a 10–20 per cent aqueous solution of hydrofluoric acid contained in a capacious leaden bowl, until it acquires a fine matt surface. To increase the brilliance of the etching the specimen is then dipped for a few seconds in concentrated hydrochloric acid. If necessary the etching is repeated.

Flick simplified this method by using suitable proportions of the two acids in one solution, thus—

Distilled water	90 c.c.
Hydrofluoric acid	10 c.c.
Hydrochloric acid	15 c.c.

For macro-etching a 10–20 per cent aqueous solution of caustic soda may also be used.

For determining the distribution of carbides, sulphides, and phosphides in cast aluminium, Czochralski (2) employs a printing process similar in character to Baumann's sulphur-printing for iron and steel (*cf.* page 68). A piece of photographic silver-paper moistened with water is placed in contact with the surface of the specimen for about 10 minutes. The gases evolved darken the paper in those places where the above mentioned impurities occur.

Preparation of Micro-sections. The grinding and polishing of aluminium and its alloys for microscopic purposes is a difficult task, requiring great experience and skill. Strict attention must be paid to the following three points if a good surface is to be obtained.

(*a*) When cutting out the specimen with a saw, it is necessary to use a sharp, fine-toothed blade and to cut with the least possible pressure, so as to avoid severe cold-working. Similarly, great care must be taken when filing, grinding, and polishing, that the thickness of the cold-worked surface layer is not increased unnecessarily.

(*b*) With an unsuitable grinding technique, particles of the grinding medium are very easily caused to penetrate into the soft surface. Any emery-paper used for grinding should be moistened with a solution of paraffin in benzol, or with alcohol, paraffin-oil, etc. According to Choulant the same result is obtained if the paper is rubbed with solid paraffin.

(*c*) For the final polishing, magnesia (*cf.* page 10) and distilled water are used on a thin cloth, or even better, on plush silk. (Dix).[1] Choulant polished with "Geolin"[2] on cloth, the latter being first moistened with spirit and then with 10–20 c.c. of a 20 per cent solution of ammonium alum in water.

In order to avoid the very difficult grinding process, microtomes have come into use for preparing a flat surface which is comparatively free from cold-working, and very suitable for direct final polishing. (Lucas, Dix.)

Structure Etching. Structure etching for microscopic purposes is carried out with the same reagents as for macro-etching, but, of course, the etching time is shortened.

Solutions of Hydrofluoric Acid. Flick's solution generally gives a good result after etching for 30–90 seconds.

Water	90 c.c.
Hydrofluoric acid	90 c.c.
Hydrochloric acid	15 c.c.

When specimens are etched with hydrofluoric acid the surface becomes covered with a dark film of an unknown nature. To remove this a further treatment is required with either strong nitric acid, or strong chromic acid solution. When the hydrofluoric acid has been washed off with water, the specimen is immersed for a moment in the nitric or chromic acid, and then again washed.

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[1] Private communication.

[2] Metal polish from Fritz Schulz, junr., A.G. Leipzig, S3.

Hydrofluoric acid has a special tendency to cause pitting.

Sodium Hydroxide Solutions. Aqueous solutions with between 5 and 20 per cent sodium hydroxide are used to a great extent for general structure-etching. The grain boundaries in pure aluminium are etched up in about 1 minute by a 20 per cent solution. In those cases where iron aluminide occurs in the grain-boundaries a period of 30 seconds is usually sufficient. Even when etching with caustic soda a dark deposit is obtained on the etched surface, but it may be removed in exactly the same way as after etching with hydrofluoric acid.

According to the A.S.S.T. *Handbook* a 10 per cent sodium hydroxide solution can be used with advantage for aluminium alloys containing zinc. These alloys can also be etched with 4 per cent nitric acid in alcohol.

Hydrofluoric Acid + Nitric Acid in Glycerine. Vilella (3) states that a solution of—

Nitric acid	1 vol.
Hydrofluoric acid	1 vol.
Glycerine	3 vol.

is an excellent general reagent for aluminium alloys, because it gives very good grain-boundary etching, and at the same time many of the structural constituents present can be identified (see below).

To bring up the grain-boundaries in the best way, the method of alternate polishing and etching is of great value. The etching reagent attacks, primarily, the aluminium-rich ground-mass and, therefore, makes visible the zone-structure caused by segregation.

The action of the reagent is slow to begin with, and the specimen should, therefore, be warmed in hot water, and transferred to the etching liquid without drying. The adhering water then accelerates the beginning of the attack.

For grain-contrast etching of copper-containing alloys Dix and Keller recommend the following solution—

Hydrofluoric acid, conc.	1·0 c.c.
Hydrochloric acid, conc.	1·5 c.c
Nitric acid, conc.	2·5 c.c.
Water	95·0 c.c.

Etching time, 10–20 seconds. After etching, the specimen is washed in hot water, and dried in an air blast. If it is wiped dry the surface may easily stain.

Ferric Nitrate. Choulant recommends for the etching of aluminium alloys containing copper a solution of ferric nitrate in alcohol acidified with nitric acid. Electrolytic iron is dissolved in concentrated nitric acid up to saturation. The solution so obtained is added to ethyl alcohol in the proportion of 5 per cent. Etching time, 3–5 minutes, at room-temperature.

Etching for the Identification of the Various Structural Constituents. The metallography of the aluminium alloys is extremely complicated and, in parts, is still incompletely investigated. All the light aluminium alloys are characterized by an aluminium-rich ground-mass wherein various constituents occur as networks, dendrites, needles, globules, eutectics, etc., according to the chemical nature and concentration of the alloying elements. The form, and occasionally even the colour, of the structural constituents present, often varies with the different degrees of dispersion brought about by mechanical work or heat-treatment. The identification of these constituents is, therefore, always most easily accomplished in the cast alloys.

Dix (see Bureau of Standards (3)) has shown that a large number of the structural constituents of the aluminium alloys can be identified directly in the polished surface of the specimen, provided that the polishing is properly done. Many constituents, however, require etching for their identification. Moreover, in cases where the constituents occur in a very finely divided form, etching is essential to remove the thin layer of the ground-mass which, even with careful polishing, very easily conceals the finely dispersed phase. Under these conditions Vilella (3) recommends the method of alternate polishing and etching.

Below is given a list, taken from the A.S.S.T. *Handbook*, showing the characteristics of the most commonly occurring structural constituents when observed in a polished, unetched, surface. Reference should also be made to the identification scheme by Dix given on page 155.

TABLE X

Structural Constituent	Colour	Characteristic Form
X-constituent[1]	Lavender	Crystal skeletons
$CuAl_2$	White to pink	Needles and granules
$FeAl_3$	Lavender to purple	Needles
Al_3Mg_2	Light. Whitish	Needles and granules
$MnAl_3$	Blue grey	Needles and crystal skeletons similar to X-component
$NiAl_3$	White	Needles and granules
Si	Blue grey	Granules, needles and dendrites
Mg_2Si	Blue	Granules and crystals
Filigree[2] constituent	Interwoven light and dark parts	Lace pattern

For the identification by etching of the various constituents a large number of methods have been proposed. The following summary is based mainly on the results of Vilella, Dix, Anderson, and several others, and on information from the A.S.S.T. *Handbook* and Bureau of Standards Circular No. 346. It includes the methods most commonly used, and their effects on the various microconstituents. It may be pointed out that a correct microscopic analysis calls for very great experience. The chemical composition of the structural constituents is, in many cases, only partly known, hence some of the notations used in the following constitute only approximate information.

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[1] Complex Fe-Al-silicide.

[2] Ternary eutectic between $CuAl_2$, Mg_2Si, and aluminium-rich solid solution.

Nitric Acid in Aqueous Solution. Nitric acid is used as a 1–25 per cent solution in water, and the etching is carried out at an accurately controlled temperature of 70° C., to which temperature the specimen itself should be heated before etching. After the proper etching time (controlled by clock) the specimen is plunged rapidly into *cold* water to stop the process. It is highly important for the whole operation to be carried out with precision if comparable results are to be obtained. In English and American literature this method is described as the "nitric acid quench."

According to Hanson and Archbutt, after etching for 2–3 seconds in the 20 per cent solution, $Cu\ Al_2$ is coloured brown, while $Fe\ Al_3$ and most of the other constituents are not affected. If the etching is increased to 30 seconds (especially in a 25 per cent solution) $Cu\ Al_2$ acquires, according to Dix, a dark colour, while $Fe\ Al_3$ retains its lavender-like shade, although its boundaries are more clearly outlined. The X-constituent acquires a light grey tone.

By etching for 40 seconds the usually weak contrast between $Fe\ Al_3$ and Fe–Si[1] can be increased.

Nitric Acid in Alcohol. For etching aluminium-magnesium alloys, 1 per cent and 10 per cent solutions are used. It is stated that $Mg_2\ Al_3$ is coloured brown by the 1 per cent solution.

According to Czochralski (3) a 5–20 per cent solution of chromic acid in water is also a particularly sensitive etching reagent for showing $Mg_2\ Al_3$.

Solutions of Ferrous Sulphate. Anderson (5) states that a 5–10 per cent aqueous solution can be used to distinguish between $Cu\ Al_2$ and $Fe\ Al_3$.

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[1] This notation is used to indicate a microconstituent, of which the exact composition is as yet undecided, but which is known to consist mainly of the elements indicated. (*Translator's Note.*)

45 seconds etching in a 10 per cent, or 3 minutes in a 5 per cent solution colours $CuAl_2$ brown to black, while $FeAl_3$ is not changed. The same result is obtained, however, with the nitric acid quench, which is more generally used.

According to Choulant, ferrous sulphate gives good results with cast alloys; but not always with worked materials, in which the size of the particles is smaller, so the following is recommended.

Ferric Nitrate in Ethyl Alcohol. Choulant's etching reagent (*cf.* page 147) colours $CuAl_2$ a light or dark brown in 3–5 minutes, and all the other compounds (including $NiAl_3$ and $FeAl_3$) are left uncoloured. To identify $FeAl_3$ the specimen is subsequently etched with 20 per cent H_2SO_4, according to Meyer's method (*cf.* page 151).

Solutions of Sodium Hydroxide. Sodium hydroxide is employed in 0·1–20 per cent aqueous solutions; the addition of alcohol retards the etching.

Light etching in 10–20 per cent aqueous solution increases the contrast between $FeAl_3$ and aluminium by bringing up the boundary-lines. In this manner a 20 per cent solution is useful for detecting $FeAl_3$ in wrought aluminium.

Caustic soda colours $CuAl_2$ dark brown after sufficiently prolonged etching. $FeAl_3$ is attacked more rapidly when it occurs together with $CuAl_2$ than when it is found alone. $NiAl_3$ is coloured brown, and is attacked more rapidly than $CuAl_2$. When $CuAl_2$, $FeAl_3$, $NiAl_3$, and $MnAl_3$, occur together in an alloy the $CuAl_2$ can be distinguished from the three last named constituents, but they, however, are all attacked in the same way and, therefore, cannot be differentiated among themselves.

Mg_2Al_3 is practically unattacked by caustic soda solution.

$MnAl_3$ is darkened by caustic soda to which some hydrogen peroxide has been added.

Dilute solutions, e.g. 0·1–1 per cent, may be used to indicate $Fe\,Al_3$ in silicon-aluminium alloys. On swabbing with such reagents the $Fe\,Al_3$ at first acquires a paler shade. With prolonged treatment this colour finally changes to brown. To distinguish between $Fe\,Al_3$ and Si the etching is stopped when the former has acquired a pronounced lavender colour.

According to Dix, after 10 seconds swabbing with 1 per cent NaOH solution, $Mn\,Al_3$ can be distinguished from Fe-Cu and Cu-Ni because it is coloured blue to brown, while the other constituents are unaffected. With a 5 seconds etch in a 10 per cent solution at 70° C., Cu-Ni can be differentiated from Fe-Cu, because the former is not attacked to any notable extent, while the latter is coloured a light brown; etch pits are formed during the attack.

Picric Acid. 4 per cent picric acid is used by A. Meyer for identifying $Cu\,Al_2$ in Al-Fe and Al-Cu alloys, and also in Al-Cu-Zn, Al-Cu-Sn, Al-Si and Al-Ni alloys containing small quantities of iron. After 10–20 minutes etching the $Cu\,Al_2$ is darkened while $Fe\,Al_3$ and other constituents are unattacked. As compared with NaOH solutions, picric acid has the advantage that the surface as a whole is scarcely attacked. On the contrary, etching with caustic soda produces a general roughening of the surface.

Sulphuric Acid. According to A. Meyer, a 10 per cent solution in water may be used at 70° C. The specimen is immersed for a few seconds in the hot solution and then cooled off in water. The cooling is, however, not so essential as it is in the "nitric acid quench."

By etching with sulphuric acid $Fe\,Al_3$ is darkened, while $Cu\,Al_2$ and $Ni\,Al_3$ are unattacked.

Dix reports that, as a result of a 30 seconds etch in a 20 per cent solution at 70° C., $Ni\,Al_3$ and Fe-Mn can be differentiated from Mn-Si and Cu-Fe-Mn. The former are not attacked, while the latter are either attacked or coloured. In a similar manner Mn Al_3, Fe-Cu, and Cu-Ni may be distinguished from Fe Al_3 and Fe-Si.

Hydrofluoric Acid. Hydrofluoric acid is used in aqueous solutions containing 0·01–10 per cent of acid. The dilute solutions can be used safely in glass dishes but, in such cases, should be freshly prepared. The strong acid should, of course, be measured out in a paraffined graduated-cylinder.

According to Dix the best method of etching for the first preliminary investigation of an aluminium alloy consists, in general, of a brief swabbing with a 0·5 per cent solution. In this way the aluminium-rich groundmass is only slightly attacked, but just sufficient to remove the thin flowed layer which, as a rule, partly covers the various constituents after polishing.

As regards the etching technique, the specimen is warmed up by washing with hot water and then swabbed with a piece of cotton-wool saturated with the etchant. After swabbing for the proper time, the specimen is washed again in hot water and dried in an air blast. Rubbing removes the products of the reactions. If swabbing is not employed the surface of the specimen darkens. This can be avoided by adding a few drops of nitric acid to the etching solution, or by momentarily dipping the specimen in strong nitric after etching. For the photomicrography of highly dispersed constituents at high magnifications very dilute solutions, e.g. 0·01–0·1 per cent, are employed.

Light etching with solutions containing 0·1–1·0 per cent HF only accentuates the intrinsic colours of the constituents, and Dix makes extensive use of such etching in his scheme of identification which is given on page 155. Prolonged etching slowly darkens Fe Al_3.

Five seconds etching in a 2 per cent solution gives Fe Al_3 a purple to pale brown colour, the X-constituent turns golden brown, and Si dark purple. HF of this concentration is of considerable value for etching up the boundary lines of the intermetallic compounds existing in the aluminium-rich ground-mass, and, as a rule, for this purpose only a momentary immersion in the etching reagent is required.

A 5 per cent solution colours $Ni\ Al_3$ brown, and blackens $Mn\ Al_3$, while a 10 per cent solution very rapidly blackens $Fe\ Al_3$ and $Cu\ Al_2$.

Solution of Hydrofluoric Acid + Nitric Acid in Glycerine. Vilella's etching reagent for aluminium alloys—

Nitric acid	1 vol.
Hydrofluoric acid	2 vol.
Glycerine	3 vol.

is, as mentioned previously, excellent for bringing out the general structure, but, at the same time, it also provides a means of identifying a number of the structural constituents which commonly occur. The reagent mainly etches the aluminium-rich ground-mass, but while some of the intermetallic compounds are not attacked and remain in their natural colours, others are etched lightly so that characteristic tints are obtained.

With this reagent, just as with all the others previously described, the fact remains that an identification of the structural constituents based only on differences of colour produced by etching is not always absolutely reliable, because variations in size of the particles of the constituents sometimes cause definite changes of colours.

The following is given according to Vilella (3).

Occurrence of Iron and Its Identification. Iron occurs in aluminium alloys in at least six different forms, or seven if a solid solution of iron in aluminium containing less than 0·13 per cent Fe is presumed to exist.

1. As a eutectic of $Fe\ Al_3$ and Al in alloys containing up to 2·5 per cent Fe. In this form it is not attacked by Vilella's etching reagent, and may be recognized by

the typical eutectic structure, and by its natural light purplish colour.

2. As star-like primary crystals of $FeAl_3$ in alloys with more than 2·5 per cent Fe. These are not attacked by the reagent but appear to be light blue-grey in colour.

3. As the X-constituent in alloys containing over 0·30 per cent Si and 0·30 per cent Fe. This constituent is formed more easily in alloys containing more than 2 per cent Cu, and there occurs associated with $CuAl_2$. It can be easily identified by its characteristic appearance, and its reddish-brown colour.

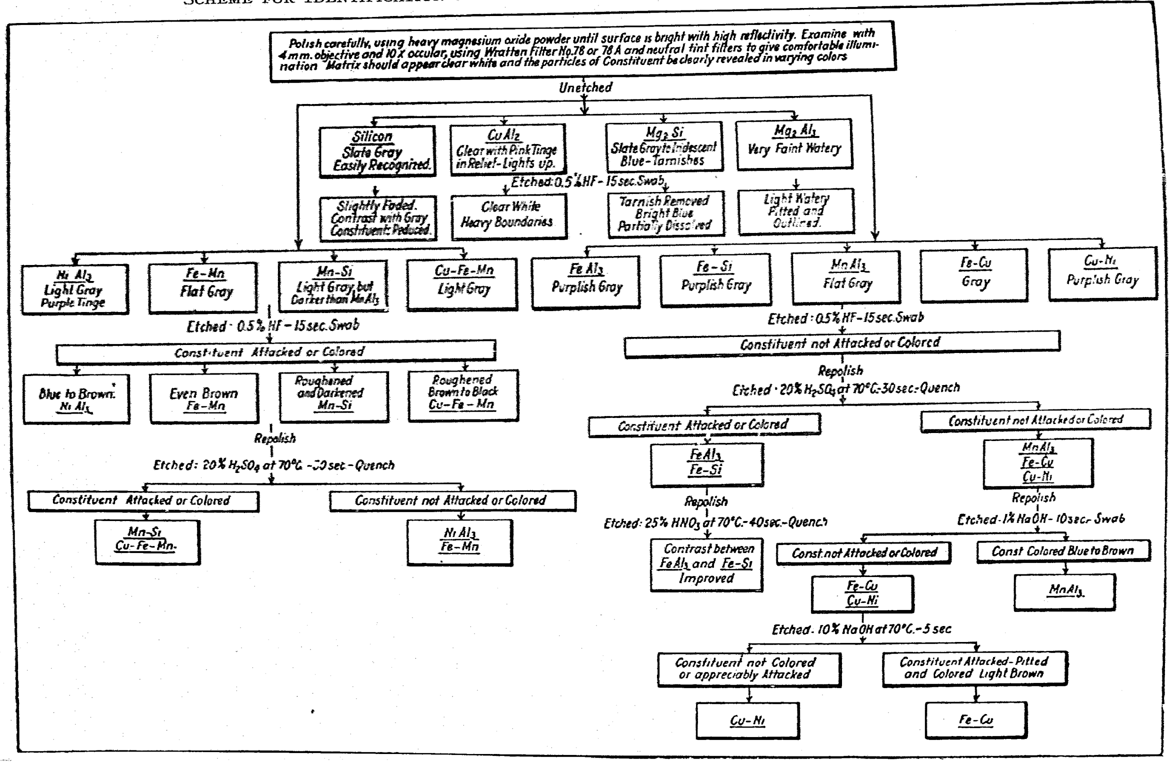
4. In alloys with 0·30 per cent Si and 1 per cent Cu, iron occurs also in the constituent which is described as "Chinese script." After etching with the glycerine solution this component is coloured deep violet when it occurs in small areas, and varies between green and red when in large areas. While the X-constituent appears in the peripheral parts of the crystal grains, the "Chinese script" is found always at the kernel of the grain, and is never associated with either the X-constituent or $CuAl_2$.

5. In alloys containing manganese, the iron appears to form a complex aluminide with the manganese. This constituent, which is water-blue in colour, is not attacked by the reagent. Duplex crystals of this aluminide often occur, with one part coloured grey, and the other the usual blue shade.

6. In alloys with a high percentage of silicon and more than 1 per cent Fe, the latter occurs as long thin needles. These act as centres of crystallization for the silicon, and are enclosed in a shell of the latter. The size of the needles, but not their number, increases with increasing iron-content. In those cases where the needles are sectioned through the core during the preparation of the specimen, the part containing iron appears with a blue colour similar to that found in material containing "Chinese script."

TABLE XI

SCHEME FOR IDENTIFICATION OF CONSTITUENTS IN ALUMINIUM AND ITS ALLOYS (DIX).


Polish carefully, using heavy magnesium oxide powder until surface is bright with high reflectivity. Examine with 4 mm. objective and 10 X occular, using Wratten Filter No. 78 or 78 A and neutral tint filters to give comfortable illumination. Matrix should appear clear white and the particles of Constituent be clearly revealed in varying colors
Unetched
Silicon Slate Gray Easily Recognized.
CuAl2 Clear with Pink Tinge in Relief-Lights up.
Mg2Si Slate Gray to Iridescent Blue-Tarnishes
Mg2Al3 Very Faint Watery
Etched: 0.5% HF - 15 sec. Swab
Slightly Faded. Contrast with Gray Constituent Reduced.
Clear White Heavy Boundaries
Tarnish Removed Bright Blue Partially Dissolved
Light Watery Pitted and Outlined.
NiAl3 Light Gray Purple Tinge
Fe-Mn Flat Gray
Mn-Si Light Gray but Darker than MnAl3
Cu-Fe-Mn Light Gray
FeAl3 Purplish Gray
Fe-Si Purplish Gray
MnAl3 Flat Gray
Fe-Cu Gray
Cu-Ni Purplish Gray
Etched · 0.5% HF - 15 sec. Swab
Constituent Attacked or Colored
Blue to Brown.* NiAl3
Even Brown Fe-Mn
Roughened and Darkened Mn-Si
Roughened Brown to Black Cu-Fe-Mn
Repolish
Etched: 20% H2SO4 at 70°C. -30 sec. - Quench
Constituent Attacked or Colored
Mn-Si Cu-Fe-Mn.
Constituent not Attacked or Colored
NiAl3 Fe-Mn
Etched: 0.5% HF - 15 sec. Swab
Constituent not Attacked or Colored
Repolish
Etched · 20% H2SO4 at 70°C.-30 sec.-Quench
Constituent Attacked or Colored
FeAl3 Fe-Si
Repolish
Etched: 25% HNO3 at 70°C.-40 sec.-Quench
Contrast between FeAl3 and Fe-Si Improved
Constituent not Attacked or Colored
MnAl3 Fe-Cu Cu-Ni
Repolish
Etched - 1% NaOH - 10 sec. - Swab
Const. not Attacked or Colored
Fe-Cu Cu-Ni
Const. Colored Blue to Brown
MnAl3
Etched - 10% NaOH at 70°C.-5 sec
Constituent not Colored or appreciably Attacked
Cu-Ni
Constituent Attacked-Pitted and Colored Light Brown
Fe-Cu

Occurrence and Identification of Copper. In concentrations of less than 2 per cent copper remains in solid-solution in the light aluminium alloys. Whatever is present over this amount appears as $Cu\,Al_2$. The copper aluminide, which is not attacked by the glycerine reagent, retains its natural pink colour. It is always found at the grain-boundaries in that part of the solid-solution which is richest in copper.

LEAD, TIN, AND BEARING METALS

The soft metals, lead and tin, are extremely difficult to prepare in a satisfactory manner for microscopic investigation. Baucke recommends grinding with a rotary movement on emery paper moistened with a mineral oil, and finally polishing with magnesia and petroleum on chamois-leather. However, the best preparation for these soft metals is undoubtedly obtained with the help of a microtome. Ordinary blotting-paper without any kind of polishing medium is sometimes recommended for the final operation.

Hydrochloric Acid. Hydrochloric acid (sp. gr. 1·12) is one of the commonest etching reagents for lead, tin, antimony, bismuth, and alloys of these metals. Wust reports an etching period of 1–10 minutes for lead. When etching alloys in strong HCl, a few seconds are often sufficient.

Hydrochloric acid gives, in general, grain-boundary etching.

The A.S.T.M. (3) recommends a 5 per cent aqueous solution for alloys containing zinc, and 5 per cent in alcohol for tin.

In "Hütte" a 1 per cent solution in alcohol is given as an etchant for lead and tin, and for white metals in general.

Nitric Acid. The A.S.T.M. recommends a 2 per cent alcoholic solution for tin-rich alloys, and a 5 per cent solution for lead-rich alloys. Meyer gives 2–3 seconds as the etching time for tin in a 2·5 per cent solution in alcohol.

Baucke prescribes for lead an etching time of 1 hour in 5 per cent alcoholic solution; more highly concentrated solutions easily produce etch pits.

Beckinsale and Waterhouse recommend a 10 per cent aqueous solution for lead.

Campbell states that care is required when etching zinc-base bearing metals with nitric acid, because they are easily over-etched.

When using aqueous solutions of nitric acid a little chromic acid may sometimes be added.

Nitric acid generally gives a grain-contrast etching.

Hydrochloric Acid | Nitric Acid. Hargreaves gives the following as an etchant for eutectic lead-tin alloys—

Hydrochloric acid	10 c.c.
Nitric acid	5 c.c.
Alcohol	85 c.c.

Hydrochloric Acid | Ferric Chloride. The A.S.S.T. *Handbook* quotes, as a good etching reagent for lead-antimony alloys, and Babbit metal—

Ferric chloride	10 grams.
Hydrochloric acid	30 c.c.
Water	120 c.c.

Hydrochloric Acid | Potassium Chlorate. Stated by Wust to give grain-contrast etching in the case of tin.

Stannous Chloride. According to *Metalltechnischer Kalender*, stannous chloride in acid solution can be used for tin and tin-rich alloys.

Acetic Acid. A 5 per cent solution in alcohol gives, according to Czochralski, grain-contrast etching in lead, although the action is very slow. For lead-sodium alloys the same author uses acetic acid + perhydrol.

Rutherford states that lead and its alloys may be etched with—

Acetic acid, glacial	3 parts
Hydrogen peroxide (9%)	1 part

Etching time, 10–30 minutes. The specimen is dried with alcohol and cleaned in strong nitric acid. The acid is removed by rapidly plunging the specimen into a large volume of water. For pure lead and pure lead-tin alloys, this treatment with nitric acid is often unnecessary.

Ammonium Persulphate + Tartaric Acid. The following is given by "Hütte"—

5 vols. of 10 per cent solution of ammonium-persulphate in water. 2 vols. of a solution of 41·4 grams tartaric acid in 150 c.c. water.

Sodium Hydroxide. In *Metalltechnischer Kalender* a saturated solution of NaOH is given for Pb-Sn alloys.

Bromine Water. Also mentioned by the above for Pb-Sn alloys.

Silver Nitrate. According to the A.S.S.T. *Handbook*, a 5 per cent solution in water is suitable for alloys of lead, tin, antimony, bismuth, and anti-friction alloys.

Picric Acid. Osmond states that picric acid dissolved in acetone can be used for etching lead and its alloys.

Electrolytic Etching. According to *Metalltechnischer Kalender* a solution of lead nitrate can be used for the electrolytic etching of lead-tin alloys.

For a similar method of etching lead Beckinsale and Waterhouse state that ammonium nitrate or acetate can be employed.

Hughes etches lead electrolytically in the following solution—

Perchloric acid (sp. gr., 1·2)	30 c.c.
Water	270 c.c.

Current density, 9 amps/dm^2 at 2 volts, with electrodes 4·5 cm. apart. Time of etching, 5–10 seconds.

To bring up etch-figures the time is prolonged up to 20 minutes. Of course, the perchloric acid solution alone etches the lead, but the process is quicker and more reliable when electrolysis is used.

ZINC AND ZINC ALLOYS

The polishing of zinc specimens is sometimes a matter of difficulty. Hayes polishes with metal-polish on chamois leather without using water. Bingham employs MgO on cloth, and states that no difficulty is experienced if, towards the end of the operation, the polishing powder is washed away, so that the final stage is carried out on an almost clean cloth. For preparing specimens by alternate polishing and etching the following reagent is recommended.

Chromic Acid | Sodium Sulphate. The Palmerton etching reagent.[1]

Chromic anhydride	10 grams.
Sodium sulphate . . .	1·5 gram.
Water	100 c.c.

This is recommended by Pulsifer. Etching for 20 seconds gives a good result with the rolled material. Cast zinc requires a somewhat longer time.

The A.S.S.T. *Handbook* advocates the above reagent with a double quantity of chromic anhydride—

Chromic anhydride	20 grams.
Sodium sulphate	1·5 grams.
Water . .	100 c.c.

Nitric Acid | Chromic Acid. Timoféefs reagent is recommended by the A.S.T.M. (3).

Nitric acid	94%
Chromic acid	6%

A few drops of this stock solution are added to 100 c.c. water immediately before use.

In "Hütte" a slight modification of the above is described—

Nitric acid	32 c.c.
Chromic acid	0·206 gram.
Water	1 c.c.

Make up with water to 1,000 c.c.

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[1] *Cf.* Pierce, W. M., *J. Inst. Metals*, 1921, *26*, 316. (Discussion on Ingall's paper.) (*Translator's note.*)

According to Desch, Timoféef's reagent is excellent for bringing up small amounts of other micro-constituents in zinc.

Nitric Acid. Hayes etches in fuming nitric acid (sp. gr. 1·5) for about 1 second. The specimen is held with zinc tongs and washed with water immediately after etching, then dried with hot air.

"Hütte" gives three different concentrations of dilute nitric acid as being suitable for zinc, namely, 0·5, 1·0, and 10 per cent solutions.

Hydrochloric Acid. The A.S.S.T. *Handbook* recommends 5 per cent hydrochloric acid for etching zinc-rich alloys.

Sodium Hydroxide. Sodium hydroxide solution is mentioned in *Chemical and Metallurgical Engineering* (see Anon (1)) as an etching reagent for zinc and zinc-rich alloys.

Iodine Solution. According to the above authority a 1 per cent solution of iodine in alcohol can be used for both zinc-rich and cadmium-rich alloys.

In the A.S.S.T. *Handbook* the following solution is given—

Iodine	1 part
Potassium iodide	3 parts
Water	10 parts

Electrolytic Etching. For ternary alloys comprising zinc, copper, and aluminium, Bingham recommends electrolytic etching in a solution of—

Hydrochloric acid	20 drops
Ethyl alcohol	50 c.c.

"Atmospheric Etching." Bingham reports that an excellent grain-boundary etching of zinc is obtained if the polished specimen is allowed to remain in the air for from a few hours up to three days. Slightly warming the specimen hastens the process. Thus, at 100° C., an etching period of 15 minutes is usually sufficient.

Hayes states that "atmospheric etching" is satisfactory for cast material but not for rolled.

SILVER AND SILVER ALLOYS

Pure silver and silver-rich alloys are, on account of their considerable softness, in many cases difficult to polish. Sometimes polishing with "metal polish" on chamois-leather gives a useful result. The best effect, however, is obtained by polishing with MgO on cloth, but it is of the greatest importance to maintain the correct amount of moisture on the polishing disc. Norbury recommends polishing with "Silvo" on a rotating "Selvyt" pad.

There is no detailed information in the literature about the etching of silver. The following etching reagents have been proposed

Etching Reagent		*Authority*
Ammonium persulphate		Anon. (1)
Nitric acid		Anon. (1)
Nitric acid	1 vol.	*A.S.S.T. Handbook*
Hydrochloric acid	5 vol.	
Water	6 vol.	
Glacial acetic acid in alcohol		*Metalltechn. Kalender*
Iodine solution + HCl		*Metalltechn. Kalender*
Ammonia + hydrogen peroxide		*Metalltechn. Kalender*
Tarnishing		*Metalltechn. Kalender*
Electrolysis in 10% citric acid		Adcock (1)
Chromic acid + hydrogen peroxide		Smith, Miller
Chromic acid + sulphuric acid.		Norbury

With reference to the last mentioned reagent, enough chromic acid is dissolved in water to make a faintly pink solution, and a few drops of sulphuric acid are then added. If too much chromic acid is present the specimen becomes covered with a film of silver chromate, while with too little the sulphuric acid produces a yellow-stained etching.

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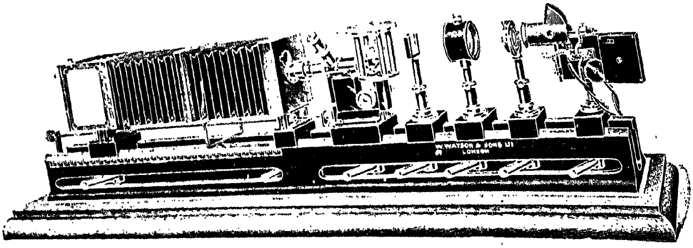

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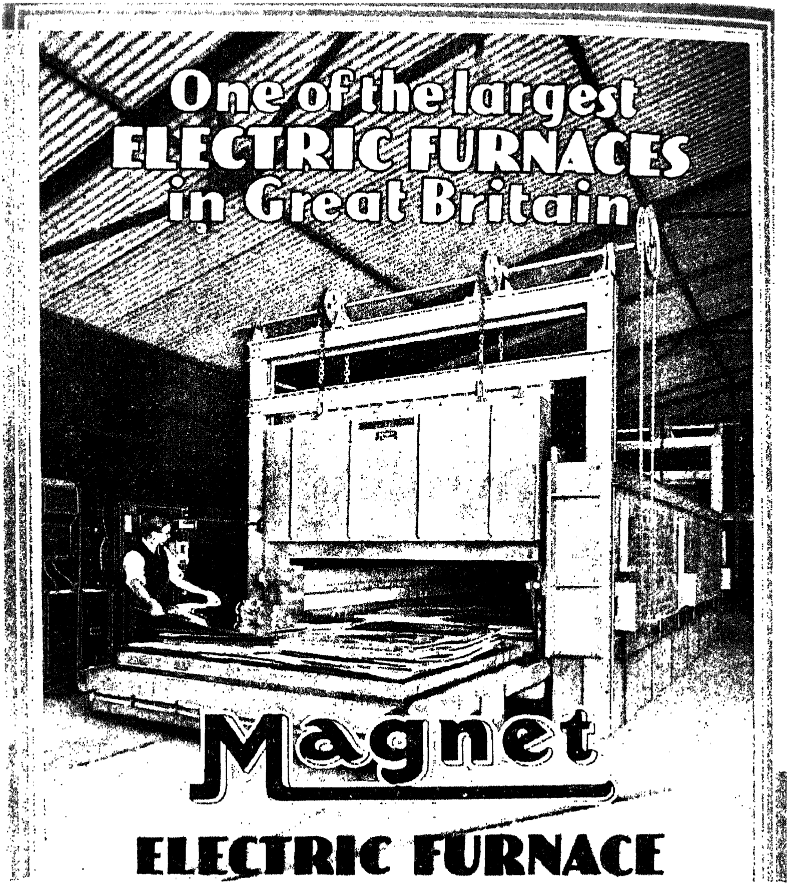

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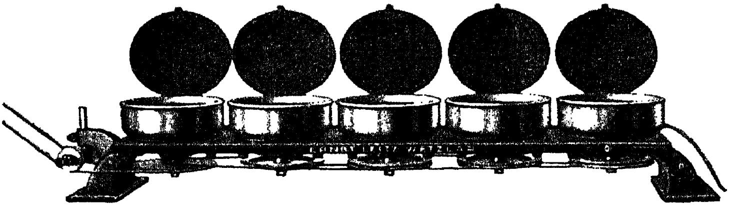

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